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2

# A COMPARATIVE EVALUATION OF TWO EXTRACTION PROCEDURES: THE TCLP AND THE EP

by

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13. ABSTRACT (Maximum 200 words) <p>The 1984 amendments to the Resource Conservation and Recovery Act (RCRA) require that the U.S. Environmental Protection Agency (EPA) restrict the land disposal of hazardous wastes. The EPA has identified four characteristics that could be used to classify a waste as hazardous: corrosivity, ignitability, reactivity, and toxicity. A waste exhibiting any one of these properties is classified as hazardous.</p> <p>The Extraction Procedure Toxicity Characteristic (EP) test is used to determine if a waste poses an unacceptable risk to ground water if improperly managed and therefore should be managed as a hazardous waste. Regulatory thresholds, based on the EP test, have been established for eight metals, four pesticides, and two herbicides.</p> <p>The Toxicity Characteristic Leaching Procedure (TCLP EPA Method 1311) was developed to address a Congressional mandate to identify additional characteristics of wastes, primarily organic constituents that may pose a threat to the</p> <p style="text-align: right;">(Continued)</p>				
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environment. The TCLP has been promulgated for use in determining specific treatment standards associated with the land disposal restrictions of RCRA. The TCLP has also been proposed as a replacement procedure for the EP test. Using the TCLP procedure, the EPA has also proposed to expand with hazardous waste regulatory levels the list of contaminants from the 14 listed in the EP protocol to a total of 52. The additional contaminants include 20 volatile organics, 16 semivolatile organics, and 2 pesticides.

The purpose of this study was to compare the results of the TCLP with those of the EP. The study was divided into three substudies. In the first substudy, a synthetic heavy metal waste was chemically solidified/stabilized, and a variety of interfering compounds were added to the solidified/stabilized waste. The solidified/stabilized waste was cured for 28 days and subjected to the TCLP and EP extractions. The extracts were analyzed for Cd, Cr, Ni, and Hg. In the second substudy, two heavy metal synthetic wastes and a perchloroethene still-bottom waste were used. The two synthetic heavy metal wastes were chemically solidified/stabilized, and the perchloroethene waste was untreated. Twelve volatile organic compounds were added to each waste type at two ratios. The EP and the TCLP extractions were performed on three samples from each waste type. The extract from each sample was analyzed for As, Ag, Ba, Cd, Cu, Ni, Pb, and Zn and the 12 volatile organic compounds. In the third substudy, volatile losses due to the mechanics of the TCLP and EP extractions were investigated, by spiking the TCLP and EP extracts with known concentrations of organic compounds. The results of this study indicate that, for most of the metal contaminants, the TCLP and EP produce similar results when TCLP extraction fluid 2 is used but differ when TCLP extraction fluid 1 is used. The results of testing for volatile organic contaminants indicate that, for 8 of the 12 contaminants, the concentrations measured in the TCLP leachates were significantly greater than those measured in the EP leachates.

14. (Concluded).

EP	Solidification
Extraction	Stabilization
Extraction Procedure	TCLP
Fixation	Toxicity Characteristic
Heavy metals	Leaching Procedure
Metals	Volatile organic compounds

## PREFACE

The study reported herein was conducted by personnel of the Environmental Laboratory (EL) of the U.S. Army Engineer Waterways Experiment Station (WES). The research was sponsored by the U.S. Environmental Protection Agency (EPA) Office of Research and Development under Interagency Agreement No. DA930146-01-05. The EPA Project Officers were Messrs. Carlton Wiles and Paul de Percin. Special assistance was provided by Mr. David Friedman of the EPA Office of Solid Waste.

The report was prepared by Mr. R. Mark Bricka, Ms. Teresa T. Holmes, and Dr. M. John Cullinane of the Water Supply and Waste Treatment Group (WSWTG), Environmental Engineering Division (EED), EL. Chemical analyses were performed by the Analytical Laboratory Group, EED. Technician support was provided by Messrs. Jim Ball, Dan Williams, and Larry L. Pugh.

Direct supervision was provided by Mr. Norman R. Francingues, Jr., Chief, WSWG. General supervision was provided by Dr. Raymond L. Montgomery, Chief, EED, and Dr. John Harrison, Director, EL.

At the time of publication of this report, Director of WES was Dr. Robert W. Whalin. Commander was COL Leonard G. Hassell, EN.

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## LIST OF ABBREVIATIONS AND SYMBOLS

### Abbreviations

ANOVA	-- analysis of variance
AVMFT	-- analysis of the variance multifactor factorial test
BDAT	-- best demonstrated available technology
EP	-- Extraction Procedure Toxicity Test
EPTC	-- Extraction Procedure Toxicity Characteristic
HDPE	-- high density polyethylene
LD <sub>50</sub>	-- lethal dose to 50 percent of the population
PCE	-- perchloroethene
PTFE	-- polytetrafluoroethylene
QA/QC	-- quality assurance/quality control
RCRA	-- Resource Conservation and Recovery Act
SIP	-- Structural Integrity Procedure
S/S	-- solidification/stabilization
TCLP	-- Toxicity Characteristic Leaching Procedure (EPA Method 1311)
ZHE	-- Zero-headspace extraction

### Symbols

B	-- weight fraction of raw waste in the solidified/stabilized interfered waste mixture
EC	-- contaminant concentration measured in the TCLP or EP extract, mg/l
EC <sub>n</sub>	-- normalized extract concentration, mg/kg
M	-- solids concentration of the solidified/stabilized waste extracted, expressed as a decimal
V	-- volume of extraction fluid, liters
W	-- weight of the wet waste extracted, kg

CONVERSION FACTORS, NON-SI TO SI  
UNITS OF MEASUREMENT

Non-SI units of measurement used in this report can be converted to SI units as follows:

<u>Multiply</u>	<u>By</u>	<u>To Obtain</u>
gallons (US liquid)	3.785412	liters
horsepower (550 foot-pounds (force) per second)	745.6999	watts
pounds (force) per square inch	6.894757	kilopascals
pounds (mass)	0.4535924	kilograms
pounds (mass) per cubic foot	16.01846	kilograms per cubic meter
pounds (mass) per gallon	0.12	kilograms per cubic decimeter

## SECTION 1

### INTRODUCTION

#### BACKGROUND

In 1976 the Congress of the United States enacted Public Law 94-580, the "Resource Conservation and Recovery Act of 1976" (RCRA). Section 3001 of the Act required that the U.S. Environmental Protection Agency (USEPA) promulgate criteria to differentiate hazardous and nonhazardous wastes (Government Institutes, Inc. 1983).

The USEPA established three methods for defining hazardous waste. First, a waste is defined as hazardous if it is listed in Table 1 of Volume 45 of the Federal Register (USEPA 1980). Second, a waste is determined to be an "Acute Hazardous Waste" if the waste is (a) found to be fatal to humans in low doses or (b) it is shown in studies to have an oral LD<sub>50</sub> (lethal dose to 50 percent of the population tested) in rats of less than 2 mg/l or a dermal LD<sub>50</sub> in rabbits of less than 200 mg (Hill 1986). Third, a waste is designated as hazardous if it exhibits a characteristic (ignitability, reactivity, corrosivity, or toxicity) of a hazardous waste as outlined in 40 CFR Part 261, Subpart C (USEPA 1987).

#### Waste Characterization

##### Definition--

The four characteristics that the USEPA established to define a nonlisted waste as a hazardous waste include: ignitability, reactivity, corrosivity, and toxicity. A waste exhibiting one or more of these characteristics is classified by the USEPA as hazardous. A waste classified as hazardous, either listed or characteristic, must be handled in accordance with Subtitle C of RCRA. This report will deal with the toxicity characteristics.

##### Toxicity--

One of the most significant dangers posed by hazardous wastes stems from the leaching of toxic constituents into ground water (Government Institutes, Inc. 1983). The USEPA's Extraction Procedure Toxicity Test (EP) addresses the properties of a waste which are directly related to the actual potential of the waste to pose a hazard to ground water. During the development of the EP, the USEPA's "primary concern was that hazardous waste might, unless subject to regulatory control, be sent to a sanitary (municipal) landfill" (Friedman 1985). Based on this concern, the EP was designed to simulate the leaching of a solid hazardous waste co-disposed with municipal waste in a sanitary landfill and to assess the potential impact of the leachate on ground-water contamination.

The toxicity characteristic is assessed using the EP. The waste is subjected to the EP, and the extract is analyzed for eight metals, four pesticides, and two herbicides. If the EP extract contains these contaminants above the limits set by the USEPA, it is determined to exhibit the toxicity characteristic and is thus a hazardous waste (USEPA 1986d). The EP is

summarized in the section below, entitled "Leaching Procedure Methods," and is presented in its entirety in Appendix A.

#### Toxicity Characteristic Leaching Procedure

The Toxicity Characteristic Leaching Procedure (TCLP) is a "second-generation" extraction procedure developed by the USEPA. The TCLP is proposed as a replacement for the EP test as a waste characterization tool. The TCLP method is summarized below in the section entitled "Leaching Procedure Methods" and is presented in its entirety in Appendix B.

Regulations defining a waste as hazardous were first promulgated in 1980. At that time, the USEPA recognized that the EP addressed only a small portion of the recognized toxic constituents (Friedman 1985). The USEPA initiated work to develop a leaching procedure that would address additional toxic constituents of hazardous waste, primarily a number of organic compounds. The TCLP has been proposed as a method of addressing the shortcomings of the EP (Friedman 1985). Since the TCLP was first published in the Federal Register (USEPA 1986a), it has undergone several modifications. This study was conducted according to the June 13, 1986, publication of the TCLP (USEPA 1986b). More recently, the November 7, 1986, version of the TCLP method has been published in the Code of Federal Regulations, Part 267, Appendix I (USEPA 1987).

#### LEACHING PROCEDURE METHODS

##### Extraction Procedure

##### Toxicity Test Method

The Extraction Procedure Toxicity Test, as outlined in USEPA's Test Methods for Evaluating Solid Waste, SW-846 (USEPA 1982), is presented in Appendix A. Specific modifications to this procedure implemented during this study are described in Section 2, "Materials and Methods." The EP extraction consists of five steps that are summarized below. A flowchart illustrating the steps in the EP is presented as Figure 1.

##### Separation Procedure--

A waste containing unbound liquid is filtered, and if the solid phase is less than 0.5% of the waste, the solid phase is discarded and the filtrate analyzed for trace elements, pesticides, and herbicides (step 5). If the waste contains more than 0.5% solids, the solid phase is extracted and the liquid phase is stored for later use.

##### Structural Integrity Procedure/ Particle Size Reduction--

Prior to extraction, the solid material must pass through a 9.5-mm standard sieve, have a surface area per gram of waste of 3.1 cm<sup>2</sup>, or, if it consists of a single piece, be subjected to the Structural Integrity Procedure. This procedure is used to demonstrate the ability of the waste to remain intact after disposal. If the waste does not meet one of these conditions, it must be ground to pass the 9.5-mm sieve.

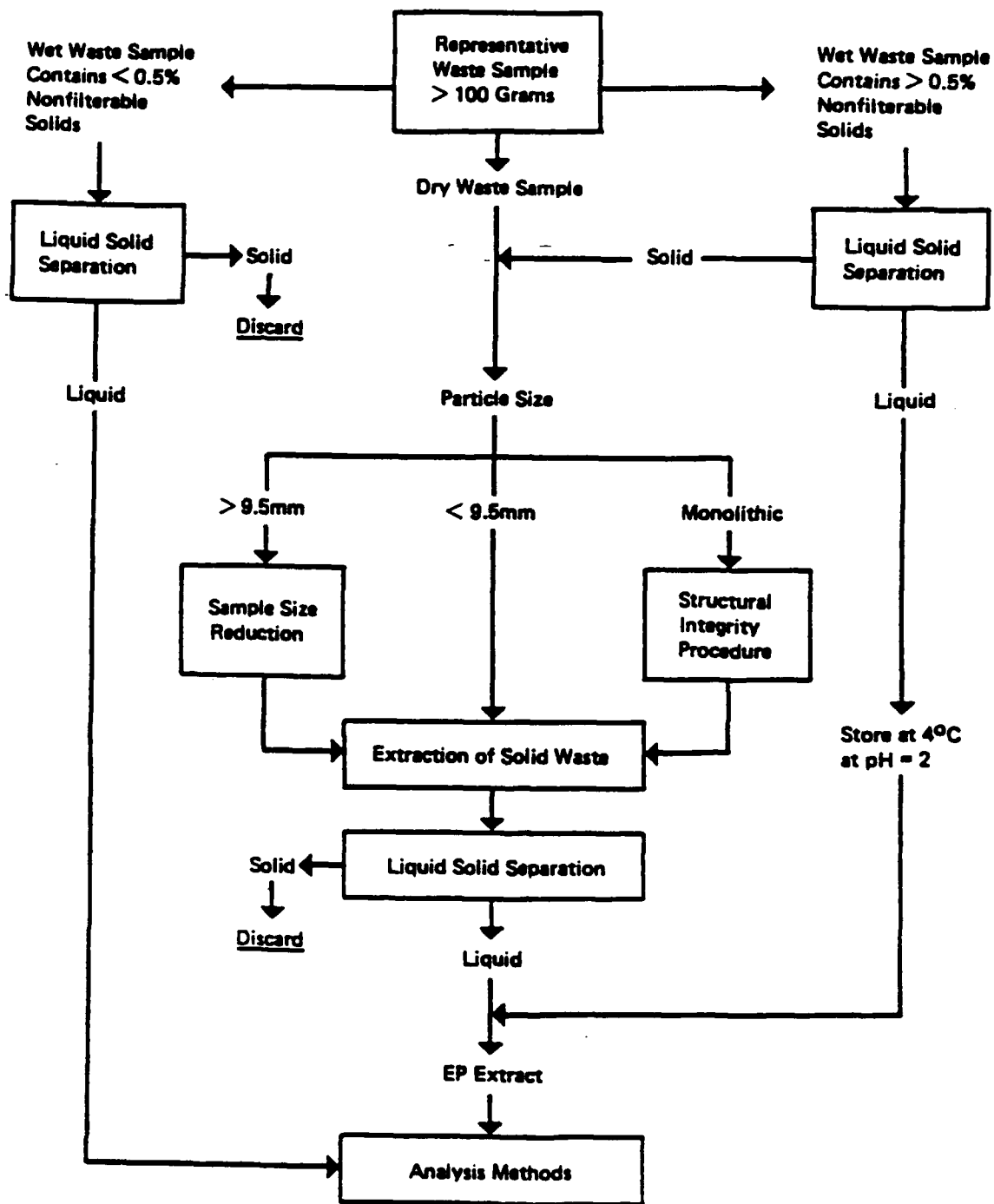


Figure 1. Extraction procedure flowchart.

## Extraction of Solid Material--

The solid material from step 2 is extracted for 24 hours in an aqueous medium whose pH is maintained at or below 5.0 using 0.5 N acetic acid. The pH is maintained either automatically or manually. (In acidifying to pH 5, no more than 4.0 ml of acid solution per gram of material being extracted may be used.)

## Final Separation of the Extraction from the Remaining Solid--

After extraction, the liquid:solid ratio is adjusted to 20:1 and the mixed solid and extraction liquid are separated by filtration. The solid is discarded and the liquid is combined with any filtrate obtained in step 1. This is the EP extract that is analyzed and compared to the threshold values listed in Table 1 (USEPA 1982).

## Testing (Analysis) of EP Extract--

Inorganic and organic species are identified and quantified using the appropriate 7000 and 8000 series of methods of analyses. These methods are listed in USEPA's manual "Test Methods for Evaluation of Solid Waste," SW-846 (USEPA 1982, 1986b)

## Toxicity Characteristic Leaching Procedure Method (EPA Method 1311)

The TCLP is conducted in two parts. The first is employed for the extraction of nonvolatile compounds; the second is employed for the extraction of volatile compounds. A flowchart illustrating the details of the TCLP is shown as Figure 2.

## Procedure When Volatiles Are Not Involved--

The TCLP for nonvolatile contaminants is a five-step procedure as described below.

Separation procedure--A waste containing unbound liquid is filtered; if the solid phase is less than 0.5% of the waste, the solid phase is discarded and the filtrate is analyzed for the desired nonvolatile contaminants. If the waste contains more than 0.5% solids, the solid phase is extracted and the liquid phase is stored for later use.

Particle size reduction--Prior to extraction, the solid material should have a particle size capable of passing a 9.5-mm standard sieve or a surface area per gram of material equal to or greater than 3.1 cm<sup>2</sup>. If the surface area is smaller than the 3.1 cm<sup>2</sup>, the particle size of the material should be reduced.

Extraction fluid determination--Prior to extraction, a small sample of the waste is tested for alkalinity. Materials with an alkalinity less than pH 5.0 are extracted using extraction fluid 1. More alkaline materials are extracted using extraction fluid 2. Extraction fluid 1 is a pH 4.93 acetic

TABLE 1. MAXIMUM CONCENTRATION OF CONTAMINANTS  
FOR CHARACTERISTIC OF EP TOXICITY

EPA Hazardous Waste Number	Contaminant	Maximum Concentration (mg/l)
D004	Arsenic	5.0
D005	Barium	100.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D012	Endrin (1,2,3,4,10,10-Hexachloro-1 7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1 4-endo, endo-5,8-dimethano-naphthalene)	0.02
D013	Lindane (1,2,3,4,5,6-Hexa-chloro-cyclohexane, gamma isomer)	0.4
D014	Methoxychlor (1,1,1-Trichloro-2,2-bis (p-methoxyphenyl)ethane)	10.0
D015	Toxaphene (C <sub>10</sub> H <sub>10</sub> C <sub>18</sub> , Technical chlorinated camphene, 67-69% chlorine)	0.5
D016	2,4-D (2,4-Dichlorophenoxyacetic acid)	
D017	2,4,5-TP (Silvex) (2,4,5- Trichlorophenoxypropionic acid)	10.0
		1.0

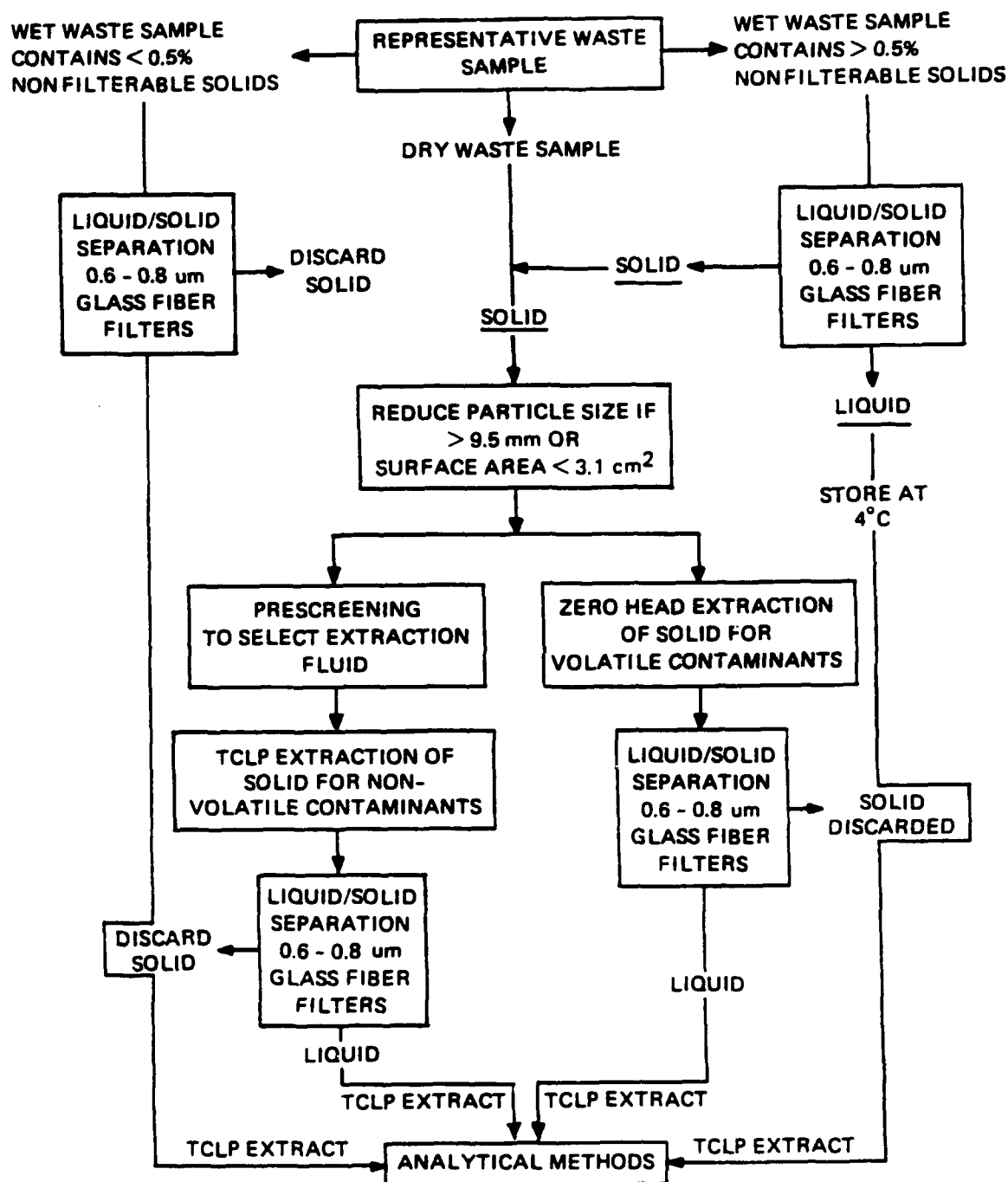


Figure 2. TCLP flowchart.

acid/sodium acetate buffer solution. Extraction fluid 2 is an acetic acid solution having a pH of 2.88.

Extraction of the solid material--The solid waste is placed in an extraction bottle, and 20 times the weight of the solid waste of the appropriate extraction fluid is used to slurry the solid waste. The waste is extracted for 18 hours.

Final separation of the extraction from the remaining solid--Following extraction, the liquid is separated from the solid by filtration. The solid is discarded, and the liquid is combined with any filtrate obtained in step 1. This is the TCLP extract that is analyzed for nonvolatile contaminants.

#### Procedure When Volatiles Are Involved--

The TCLP used for the extraction of volatile contaminants is a four-step procedure as described below. Table 2 specifies the volatile contaminants listed by the TCLP.

Separation procedure--A separation procedure, similar to the one used for the nonvolatile extraction, is performed. This procedure was described in the subsection entitled "Procedure When Volatiles Are Not Involved."

Particle size reduction--The method used to reduce the particle size of the waste extracted for volatile compounds is similar to the particle size reduction method used for the nonvolatile extraction. This method is also described under the nonvolatile section.

Zero-headspace extraction of the solid material--The solid waste is extracted utilizing extraction fluid 1 regardless of pH. The waste is placed in a zero-headspace extraction (ZHE) device and slurried (under zero head conditions) with extraction fluid at 20 times the weight of the waste. The waste is extracted for 18 hours.

Final separation of the extraction from the remaining solid--Following extraction, the liquid is simultaneously filtered and removed from the ZHE device. The solid is discarded, and the extraction liquid is combined with any filtrate obtained in step 1. This is the TCLP extract that is analyzed for volatile contaminants.

#### Comparison of EP and TCLP Methods

There are many contrasts between the EP and TCLP methods (Callaway, Parr, and Bollinger 1987), some of which are quite prominent; others are buried deep within the procedures. The most obvious difference is that the TCLP requires the use of the ZHE vessel for volatile compounds and an extraction fluid selection step for nonvolatile extractions. Other differences include:

1. In the TCLP method for nonvolatiles, one of two extraction fluids is selected to extract the solid waste sample. The type of extraction fluid is determined in an initial test on the waste and is based on the waste's alkalinity. Extraction fluid 1 is an acetate buffer at a pH of  $4.93 \pm 0.05$ . Extraction fluid 2 is an acetic acid solution

TABLE 2. VOLATILE CONTAMINANTS AS LISTED BY THE TCLP\*

1. Acetone	8. Methyl isobutyl ketone
2. n-Butyl alcohol	9. Tetrachloroethylene
3. Carbon disulfide	10. Toluene
4. Carbon tetrachloride	11. 1,1,1-Trichloroethane
5. Chlorobenzene	12. Trichloroethylene
6. Methylene chloride	13. Trichlorofluoromethane
7. Methyl ethyl ketone	14. Xylene

\* If any or all of these compounds are of concern, the zero-headspace extraction vessel shall be used. If other (nonvolatile) compounds are of concern, the conventional extraction bottle shall be used.

with a pH of  $2.88 \pm 0.05$ . The EP uses distilled deionized water as an extraction fluid, and 0.5 N acetic acid is added to the solid waste/water slurry to maintain the pH at  $5.0 \pm 0.2$ . The acetic acid is added as required, up to a maximum of 4 g of 0.5 N acetic acid per 1 g of solid waste extracted.

2. The TCLP method for volatiles requires the use of extraction fluid 1. The EP has no volatiles extraction procedure.
3. The TCLP requires that the ZHE vessel be used for volatiles extraction. Extraction bottles made of glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel are specified for organic or inorganic contaminants. High density polyethylene (HDPE), polypropylene, or polyvinyl chloride may be utilized as extraction vessels when nonvolatile compounds are extracted. The EP is vague about extraction vessel design.
4. The TCLP procedure requires the use of 0.6- to 0.8- $\mu$ m glass fiber filters and excludes the use of prefilters. The EP requires the use of 0.45- $\mu$ m cellulose triacetate filters and allows the use of glass fiber prefilters.
5. The TCLP requires that the particle size of the solid be small enough to pass a 9.5-mm standard sieve. The EP allows the use of the Structural Integrity Procedure if the sample is monolithic in nature. If the sample is not a monolith, the EP requires that the particle size be small enough to pass a 9.5-mm standard sieve.
6. The TCLP requires rotary agitation in an end-over-end fashion at  $30 \pm 2$  rpm. The EP allows the use of either a stirred open vessel or a rotary end-over-end agitator.

7. The extraction period for the TCLP is 18 hours. The extraction period for the EP is 24 hours  $\pm$  2 hours.
8. The EP requires monitoring and adjustment of the pH during the extraction. The TCLP does not.

#### ASSOCIATED PROJECTS

The waste materials utilized in this study were also used in three other studies funded by the USEPA and conducted at the U.S. Army Engineer Waterways Experiment Station. These studies include: (1) Investigation of Test Methods for Solidified Waste Characterization - A Cooperative Program," (2) "Evaluation of Factors Affecting Stabilization/Solidification of Toxic and Hazardous Waste," and (3) "Evaluation of Stabilization/Solidification as a Best Demonstrated Available Technology." Brief descriptions of these projects and their relationships to this study are presented below.

##### Investigation of Test Methods for Solidified Waste Character- ization - A Cooperative Program

This study was designed to develop and evaluate techniques to assess the effectiveness of a variety of solidification/stabilization<sup>1</sup> (S/S) technologies. Three laboratories, the U.S. Army Engineer Waterways Experiment Station (WES), the Wastewater Technology Centre (WTC), and the Alberta Environmental Centre (AEC), participated in the study. Five raw wastes were solidified/stabilized by 15 commercial S/S vendors. The resulting solidified/stabilized materials were shipped to the three labs (WES, WTC, and AEC), and 12 testing protocols were performed on the solidified/stabilized materials. Details of the cooperative study are outlined in the report entitled "Laboratory Assessment of Short-Term Test Methods for the Evaluation of Solidified/Stabilized Waste Materials" (Holmes and Bricka 1988) and in "Investigation of Test Methods for Solidified Waste Characterization: A Cooperative Program" (Stegemann and Cote, in press).

One of the five raw wastes developed for the cooperative study was a synthetic metal solution formulated by the WTC laboratory. This waste is referred to as the "WTC waste" through the remainder of this report.

##### Evaluation of Factors Affecting S/S of Toxic and Hazardous Wastes

This study (referred to as "The Interference Project") was designed to assess the effects of a variety of industrial chemicals on the physical and chemical properties of typical S/S processes.

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<sup>1</sup> Solidification/stabilization is a process that involves the mixing of a hazardous waste with a binder material to enhance the physical and chemical properties of the waste and to chemically bind any free liquid (USEPA 1986a).

Many hazardous wastes contain materials that are known to inhibit the setting and strength development properties of S/S techniques. The effects of five organic and five inorganic chemicals on a solidified/stabilized synthetic heavy metal sludge were evaluated. The synthetic metal sludge was solidified/stabilized using three generic binders. The details of this study are outlined in a report entitled "An Assessment of Materials That Interfere with Stabilization/Solidification Processes" (Cullinane, Bricka, and Francingues 1987).

The synthetic metal plating sludge evaluated in the Interference Project was also used in this TCLP/EP comparison study. The synthetic metal plating sludge is identified as the "WES waste" through the remainder of this report.

#### Evaluation of S/S as a Best Demonstrated Available Technology (BDAT)

The BDAT S/S study determined whether S/S techniques could be applied to a variety of "listed" wastes and evaluated the effects of the S/S techniques on the mobility of the contaminants contained in the wastes. Data collected as part of the BDAT S/S study are being utilized by the USEPA to support the development of treatment standards for wastes subject to the land disposal restrictions (USEPA 1987). The details of this study are outlined in a series of reports (see Bricka, Holmes, and Cullinane 1988).

One of the listed wastes evaluated in the BDAT S/S study, a by-product from the reclamation of spent perchloroethene solvent, was also used in this TCLP/EP comparison study. Throughout the remainder of this report, the perchloroethene solvent waste is identified as the "PCE waste."

#### PURPOSE AND SCOPE

The purpose of this study was to compare the results of the TCLP to those of the EP. This comparison was accomplished by dividing this study into sub-studies. The first substudy evaluated the metal-extraction effectiveness of the two methods. The second substudy investigated the extraction of volatile compounds. The third substudy examined the volatile losses due to the mechanics of conducting the extractions and the storage of extracts prior to analyses.

#### ORGANIZATION OF THE REPORT

##### Section 1: Introduction

The introduction briefly describes the origin of the EP and TCLP extractions, the difference between the TCLP and EP extractions, various projects associated with this study, and the scope of the study.

##### Sections 2 and 3:

##### Conclusions and Recommendations

Conclusions based on the results of this study and recommendations for future research are presented in these sections.

#### Section 4: Materials and Methods

This section describes the three separate substudies conducted as part of this research effort. Each substudy details the methods used for preparing the wastes and the extraction procedures performed.

#### Section 5: Results

This section presents the results of the EP and TCLP extraction and compares the extraction tests.

## SECTION 2

### CONCLUSIONS

This study was conducted to compare the results of the TCLP and the EP. The EP and TCLP extractions were performed on a number of different wastes subjected to a variety of conditions. Based on the results of this study, the following conclusions can be drawn.

(1) Generally, the TCLP was a more aggressive leaching procedure than the EP.

(a) When the TCLP extraction fluid 2 was used for the extraction of metal contaminants, the EP and TCLP produced similar results.

(b) When the TCLP extraction fluid 1 was used for the extraction of metal contaminants, the EP and TCLP produced statistically different results, with the TCLP generally being the more aggressive extraction.

(c) The TCLP zero-headspace extraction was only a slightly more aggressive extraction for volatile organics than the EP extraction in this study.

(2) Although the TCLP zero-headspace extraction was a more aggressive extraction procedure than the EP for the volatile organics, the difference in the concentrations of volatile organics in the TCLP and EP extracts was less than expected.

(3) When the ZHE vessel was used, cross contamination presented a potential problem.

(4) The TCLP and EP extraction of the solidified/stabilized specimens appeared to produce conditions that permit dechlorination reactions to occur. Significant amounts of 1,1-dichloroethene were detected in the TCLP and EP extracts although no 1,1-dichloroethene was added, and none was detected in the raw wastes.

### SECTION 3

#### RECOMMENDATIONS

The TCLP method, while more difficult to perform than the EP method, is an extraction test that can be performed in most laboratories. The TCLP method, unlike the EP method, addresses semivolatile and volatile contaminants. Several areas should be clarified in the TCLP extraction method. The following recommendations are based on the results of this study.

(1) The ZHE vessel is difficult to clean. The TCLP method needs to make recommendations on the most effective method of cleaning the ZHE vessel. Modification of the valve design is highly recommended to improve cleaning techniques.

(2) The TCLP method is vague about procedures for sample collection from the ZHE vessel when Tedlar bags are not used. A section describing the collection of a sample using volatile vials should be included in the TCLP method.

(3) Additional research should be initiated to investigate why volatile chlorinated compounds extracted from solidified/stabilized wastes are converted to other chlorinated forms.

## SECTION 4

### MATERIALS AND METHODS

#### PROJECT OVERVIEW

##### General Approach to the Investigation

This project includes two independent evaluations, Study A and Study B. These studies compare the results from the-EP and TCLP extraction procedures using common waste types. Project flowcharts for both studies are presented in Figures 3 through 5.

##### Study A--

Study A was conducted in four phases, as summarized below.

Phase I--A synthetic metal plating sludge containing cadmium (Cd), chromium (Cr), nickel (Ni), and mercury (Hg) was prepared.

Phase II--The synthetic sludge was solidified/stabilized using a lime kiln dust binding agent. Prior to the initial set, the solidified/stabilized sludge was divided into portions, and a single "interfering" compound was mixed with each portion of solidified/stabilized sludge. A total of 10 interfering compounds were added to the various portions of the sludge.

Phase III--The kiln dust/sludge/interference mixtures were cured for 28 days. After curing, each waste mixture was subjected to the EP extraction and the TCLP extraction. The extracts of the TCLP and EP were analyzed for Cd, Cr, Ni, and Hg.

Phase IV--The results of chemical analyses performed for the TCLP and EP extracts were compared to evaluate differences between the two extraction methods.

##### Study B--

Study B was conducted in four phases as summarized below.

Phase I--Three wastes, the metal sludge used in Study A, a synthetic metal waste solution, and a perchloroethene still-bottom waste (K030), were used in Study B. The synthetic metal solution and the metal sludge were solidified/stabilized using Type I Portland cement as a binding agent. The perchloroethene sludge was not solidified/stabilized. Prior to the initial set, each of these solidified/stabilized mixtures and the untreated perchloroethene waste were divided into two portions. Twelve volatile organic compounds were added to each portion at approximate concentrations of 0.1% and 1.0%, respectively.

Phase II--These six mixtures were placed in sealed bottles and allowed to cure for 14 days. After curing, each waste material was subjected to the EP and TCLP extractions. The TCLP and EP extracts were analyzed for metals and volatile organic compounds.

PROJECT: LABORATORY COMPARATIVE EVALUATION  
OF THE  
TCLP AND EP

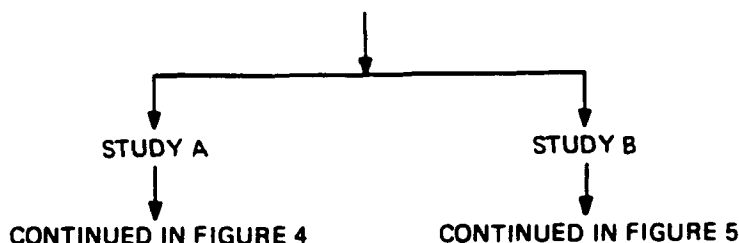


Figure 3. Project flowchart for overall study.

Phase III--The EP and TCLP extracts were spiked with known concentrations of three volatile organic compounds. The extract solutions were spiked during two steps of the EP and the TCLP methods: prior to the extraction, and after the liquid/solid separation step. These spike compounds were used to detect any volatile losses that might occur during implementation of the extraction procedure or storage of the extracts prior to chemical analysis.

Phase IV--The results of chemical analyses on the TCLP and EP extracts were compared to evaluate differences between the two extraction methods.

Wastes Selected for Study

Three wastes were selected for use in this evaluation: a synthetic metal plating sludge (WES waste), a synthetic metal plating solution (WTC waste), and a perchloroethene still-bottom waste (PCE waste). The rationale for selecting these wastes is discussed below.

WES Waste--

The WES waste was a synthetic sludge made from reagent grade chemicals. This waste contains high concentrations of toxic metals (Cd, Cr, Ni, and Hg) and was a good candidate for study because it was likely to leach the contaminants at detectable levels.

WTC Waste--

The WTC waste was prepared from reagent grade chemicals and contained high concentrations of arsenic, cadmium, chromium, and lead. Two of these metals were not found in the WES waste, therefore adding to the number of parameters evaluated by this investigation.

PCE Waste--

The PCE waste was an actual industrial waste produced as a by-product from the reclamation of spent dry cleaning solvent. It contained 14 toxic metals, including antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver, thallium, and zinc.

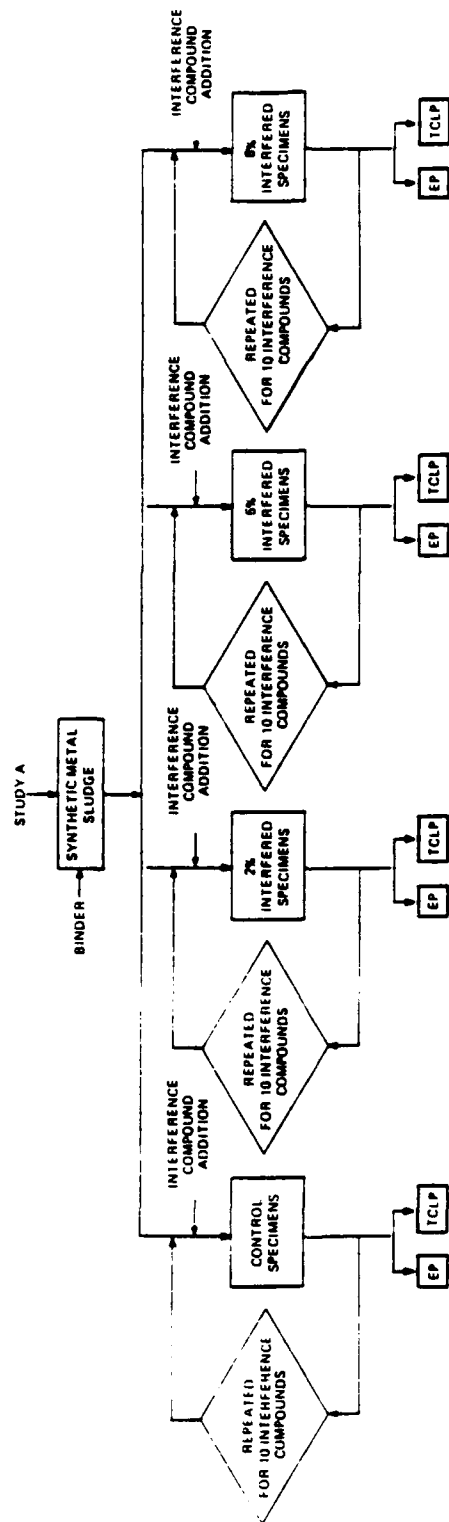


Figure 4. Project flowchart for Study A.

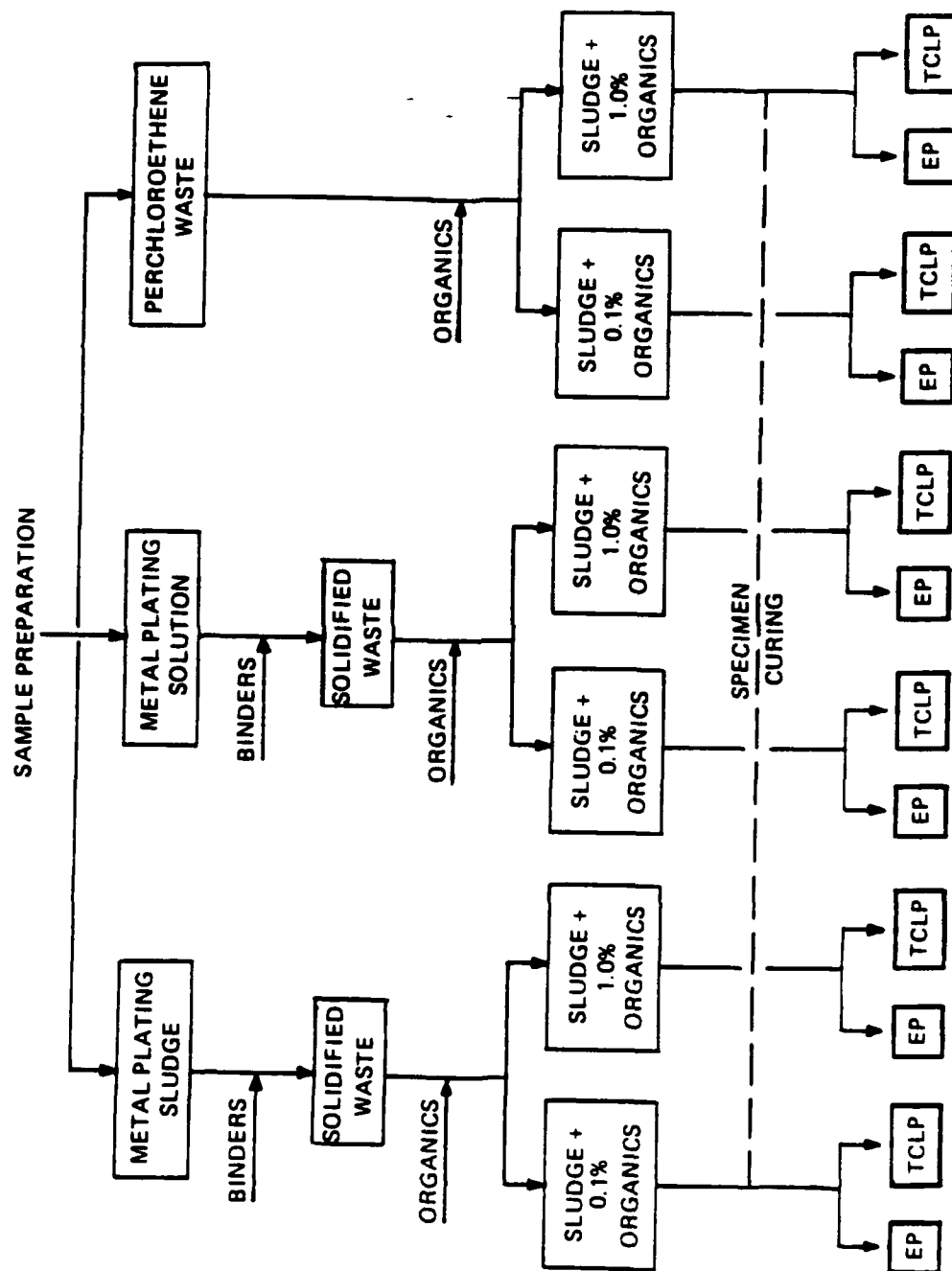


Figure 5. Project flowchart for Study B.

## STUDY A

### Waste Description

The WES sludge is a synthetic waste produced by hydroxide precipitation of a concentrated metal nitrate solution. The metal nitrate solution was prepared by dissolving four metal nitrate salts, cadmium nitrate ( $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ ), chromium nitrate ( $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ), nickelous nitrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), and mercury nitrate ( $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ ) in 500 gal\* of American Society for Testing and Materials type III water (ASTM 1986). This mixture produces a solution with metal ion concentrations approximately 600 times the EP limits. This metal nitrate solution was treated with 97.5 lb of calcium hydroxide to precipitate the metal ions from solution. The resulting sludge was separated from the supernatant, and the sludge was filtered using an Eimco Model 3613 vacuum filter. Typically, the filtration process produced a sludge with 27% to 35% solids by weight. The dewatered sludge was homogenized with a model 20-E Stow paddle type mixer and passed through a 30-mesh screen to remove large particles. A moisture analysis was performed on the homogenized sludge. The method used in determining the moisture content is outlined in Appendix C. Based on the sludge's moisture content, supernatant was added to the sludge to adjust the solids content of the sludge to  $25\% \pm 0.5$ . This 25% solids sludge was a semifluid with an approximate density of 11.7 lb/gal, and a pH of 11. Results of the average bulk chemical analyses for this sludge are presented in Table 3. This material was stored at 4° C until needed for testing.

TABLE 3. ANALYSES OF THE WES SLUDGE

Parameter	Ionic Species	Concentration (mg/kg wet weight)
Cadmium	$\text{Cd}^{+2}$	4,000
Chromium	$\text{Cr}^{+3}$	18,000
Nickel	$\text{Ni}^{+2}$	19,000
Mercury	$\text{Hg}^{+2}$	200
Calcium	$\text{Ca}^{+2}$	60,000
Total solids	--	25%

### Preparation of Test Samples

Approximately 250 lb of 25% sludge was divided into ten 25-lb samples. The sludge was solidified/stabilized using lime kiln dust. Compositional and chemical analyses of the kiln dust used in this study are summarized in

\* A table of factors for converting non-SI units of measurement to SI (metric) units is presented on page xiii.

Tables 4 and 5. Each 25-lb sample of sludge was solidified/stabilized with 27.5 lb of the lime kiln dust. Prior to the initial set, each sample was subdivided into four equal portions. One of the ten interfering compounds (Table 6) was added to each portion at approximate percentages\* of 0%, 2%, 5%, or 8% (wet weight interference compound to kiln dust/sludge mixture). Due to the large number of samples required, all the specimens used in this study could not be prepared at one time. The sludge/kiln dust/interference mixtures were prepared in several batches according to the schedule presented in Table 7.

After each waste/kiln dust/interference mixture was thoroughly homogenized, two samples were prepared by pouring the slurry into two 850-ml plastic disposable cylindrical molds. The samples were cured in the molds at 23° C and 98 percent relative humidity for a minimum of 24 hours and removed from the molds whenever they developed sufficient strength to be free standing. After removal from the molds, the samples continued curing for a period of 28 days under the same conditions.

At the end of the 28-day cure period, the samples were ground with a mortar and pestle to pass a 9.5-mm sieve. Ground materials from duplicate samples were recombined and sealed in 1,000-ml polyethylene bottles. Thus, a single sample was prepared for each of the 10 interfering compounds at the four interference compound percentages.

The bottles were agitated in an end-over-end fashion to mix their contents, and samples were collected to determine the moisture content of the materials (as outlined in Appendix C). Duplicate subsamples were collected from each bottle containing the ground materials. These duplicate subsamples were subjected to EP and TCLP methods outlined in Appendices A and B. A method blank was carried through the extraction procedures for each interference compound. The matrix of test specimens subjected to the EP and TCLP extractions along with the age of the extraction sample at the time of analysis is presented as Table 7.

#### Analytical Procedures

The EP and TCLP extracts were analyzed for various metals. The analytical and digestion methods used are presented in Table 8.

#### Quality Assurance/Quality Control

Both internal and external laboratory quality assurance/quality control (QA/QC) measures were performed during the course of Study A. External QA/QC is defined as that which is performed by the laboratory conducting the extractions; internal QA/QC is the which performed by the laboratory that analyzes the extract for the contaminants of interest. External QA/QC consisted of (1) carrying method blanks through the extractions every 9th sample and (2) submitting standards to the analytical laboratory every 10th sample. Internal QA/QC consisted of performing the metal analysis by the method of standard additions.

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\* Actual concentrations were 0%, 1.96%, 4.76%, and 7.41%.

TABLE 4. COMPOSITIONAL ANALYSES OF BINDER MATERIALS

Compositional Analysis	Type I Cement (as percent)	Flyash Class F (as percent)	Kiln Dust (as percent)
Silicon dioxide ( $\text{SiO}_2$ )	20.47	49.67	6.94
Aluminum oxide ( $\text{Al}_2\text{O}_3$ )	5.40	29.15	4.23
Iron (III) oxide ( $\text{Fe}_2\text{O}_3$ )	3.58	7.11	1.47
Calcium oxide ( $\text{CaO}$ )	64.77	1.26	62.93
Magnesium oxide ( $\text{MgO}$ )	0.87	1.43	0.44
Sulfite ( $\text{SO}_3$ )	2.73	0.23*	7.01
Insoluble residue	0.17	70.70†	3.09
Moisture loss	0.43	0.12‡	0.05
Loss on ignition	0.96	4.07	14.08
Titanium (IV) oxide ( $\text{TiO}_2$ )	0.28	0.20	0.11
Manganese oxide ( $\text{Mn}_2\text{O}_3$ )	0.06	0.00	0.00
Phosphorus pentoxide ( $\text{P}_2\text{O}_5$ )	0.28	1.00	0.05
<u>Total Alkali</u>			
Sodium oxide ( $\text{Na}_2\text{O}$ )	0.12§	0.23	0.25§
Potassium oxide ( $\text{K}_2\text{O}$ )	0.28	2.33	0.40
Sodium (Na)	0.05	0.10	0.10
Potassium (K)	0.11	0.97	0.17
Total as $\text{Na}_2\text{O}$	0.30	1.76	0.51
<u>Acid-Soluble Alkali</u>			
Sodium oxide ( $\text{Na}_2\text{O}$ )	0.12	0.06	0.25
Potassium oxide ( $\text{K}_2\text{O}$ )	0.28	0.50	0.40
Sodium (Na)	0.05	0.03	0.10
Potassium (K)	0.11	0.21	0.17
<u>Water-Soluble Alkali</u>			
Sodium oxide ( $\text{Na}_2\text{O}$ )	0.018	0.050	0.021
Potassium oxide ( $\text{K}_2\text{O}$ )	0.139	0.105	0.050
Sodium (Na)	0.0075	0.0210	0.0088
Potassium (K)	0.0577	0.0440	0.0208

\* Acid-soluble sulfate.

† Includes  $\text{SiO}_2$  (silicon dioxide).

‡ Free water.

§ Cement, lime, and kiln dust alkalies totally dissolve in acid; therefore, total acid and acid-soluble analysis will be the same.

TABLE 5. CHEMICAL ANALYSES OF BINDER MATERIALS

Chemical Analysis	Cement Type I (mg/kg)	Kiln Dust (mg/kg)	Flyash Class F (mg/kg)
Silicon (Si)	95,700	1,900	32,400
Total sulfur (S)	10,800	700	31,200
Titanium (Ti)	1,400	50	600
Phosphorus (P)	900	60	200
Antimony (Sb)	<1.77	<1.63	13.3
Arsenic (As)	13.1	14.7	172
Beryllium (Be)	2.13	4.24	28.9
Cadmium (Cd)	0.284	2.28	1.01
Chromium (Cr)	61.3	30.0	139
Copper (Cu)	14.9	12.7	196
Lead (Pb)	2.13	15.6	57.7
Mercury (Hg)	<0.100	<0.100	<0.100
Nickel (Ni)	25.9	33.6	190
Selenium (Se)	<17.7	<16.3	<19.5
Silver (Ag)	<3.54	<3.26	<3.90
Thallium (Tl)	<10.6	<9.78	13.6
Zinc (Zn)	41.8	107	211
Aluminum (Al)	23,100	13,500	150,000
Barium (Ba)	178	119	1,350
Calcium (Ca)	454,000	440,000	12,000
Cadmium (Cd)	<10.6	<9.78	77.2
Iron (Fe)	25,400	14,800	50,700
Magnesium (Mg)	5,460	3,040	6,040
Manganese (Mn)	503	64.2	156
Sodium (Na)	1,270	2,110	2,740
Tin (Sn)	195	73.0	118
Vanadium (V)	55.6	34.6	351

TABLE 6. INTERFERENCE COMPOUNDS UTILIZED IN STUDY A

Organic Interference	Inorganic Interference
Oil	Lead nitrate-Pb(NO <sub>3</sub> ) <sub>2</sub>
Grease	Zinc nitrate-Zn(NO <sub>3</sub> ) <sub>2</sub>
Hexachlorobenzene-HCB	Copper nitrate-Cu(NO <sub>3</sub> ) <sub>2</sub>
Trichloroethene-TCE	Sodium hydroxide-NaOH
Phenol	Sodium sulfate-Na <sub>2</sub> SO <sub>4</sub>

## STUDY B

Waste Description

## WES Sludge--

The WES sludge used in Study B was the same synthetic metal waste that was used in Study A. A detailed description of how this waste was prepared is given in the Study A "Waste Description" section.

## WTC Waste--

The WTC metal solution was prepared by dissolving 0.04 mole of chromium chloride (CrCl<sub>3</sub>·9H<sub>2</sub>O), cadmium nitrate (Cd(NO<sub>3</sub>)<sub>2</sub>·2H<sub>2</sub>O), lead nitrate (Pb(NO<sub>3</sub>)<sub>2</sub>), sodium arsenite (NaAsO<sub>2</sub>), and phenol in ASTM type I water (ASTM 1986). This solution had a total dissolved solids content of 3.4%, a density of 62 lb/ft<sup>3</sup>, and a pH of 2.5. Results of the bulk chemical analysis for this waste are presented in Table 9. This material was stored at 4° C until needed for testing.

## PCE Waste--

The PCE waste was generated as a by-product from the reclamation of spent dry cleaning solvent. The PCE waste is a listed hazardous waste (K030) (USEPA 1987). The waste production and reclamation process is summarized below.

Perchloroethene is typically used as a cleaning solvent in dry cleaning operations. When the PCE becomes contaminated with dirt and solids it is passed through paper cartridge filters to remove the dirt and solids and extend the useful life of the PCE. Eventually, these paper filters become fouled, and the entire cartridge must be disposed. The PCE solvent retained in the filter can be reclaimed for reuse by utilizing a batch distillation treatment method. A schematic diagram of the batch distillation unit is shown in Figure 6. The PCE waste utilized in this study was the residual, or bottoms product, resulting from this type of distillation operation. A chemical analysis of the PCE waste is presented in Table 10.

TABLE 7. TEST SPECIMEN MATRIX FOR STUDY A METALS DATA: EXTRACTION SAMPLE AGE  
AT THE TIME OF ANALYSIS

Batch No.	Interference Compound	Interference Concentration (percent)	No. of TCLP Extractions Prepared	No. of EP Extractions Prepared	Average Age of TCLP Samples at the Time of Analysis (in days)				Average Age of EP Samples at the Time of Analysis (in days)			
					Cd	Cr	Ni	Hg	Cd	Cr	Ni	Hg
1	Oil	0	2	2	42	42	47	5	42	48	48	5
		2	2	2	42	55	52	5	50	62	48	5
		5	2	2	42	62	57	5	57	55	48	5
		8	2	2	42	47	48	5	57	55	48	5
2	Grease	0	2	2	46	38	45	43	46	38	38	43
		2	2	2	46	46	51	43	46	38	47	43
		5	2	2	46	42	51	43	46	38	56	43
		8	2	2	46	46	51	43	46	38	56	43
3	Lead (Pb)	0	2	2	52	41	66	2	52	41	66	2
		2	2	2	52	41	66	2	52	56	66	2
		5	2	2	41	41	57	2	52	71	66	2
		8	2	2	52	41	57	2	47	57	66	2
4	Copper (Cu)	0	2	2	42	39	44	43	42	49	53	43
		2	2	2	42	39	44	43	42	39	53	43
		5	2	2	42	39	53	43	42	39	53	43
		8	2	2	42	39	53	43	42	49	53	43
5	Zinc (Zn)	0	2	2	46	39	39	46	46	43	39	46
		2	2	2	46	39	52	46	46	39	39	46
		5	2	2	46	39	52	46	46	39	39	46
		8	2	2	46	39	52	46	44	39	39	46
6	Sodium hydroxide (NaOH)	0	2	2	50	41	41	48	50	41	41	48
		2	2	2	50	41	41	48	50	41	61	48
		5	2	2	50	41	41	48	50	41	61	48
		8	2	2	50	41	41	48	50	41	61	48

(Continued)

TABLE 7. (Concluded)

Batch No.	Interference Compound	Interference Concentration (percent)	No. of TCLP Extractions Prepared	No. of EP Extractions Prepared	Average Age of TCLP Samples at the Time of Analysis (in days)				Average Age of EP Samples at the Time of Analysis (in days)			
					Cd	Cr	Ni	Hg	Cd	Cr	Ni	Hg
7	Sodium sulfate (Na <sub>2</sub> SO <sub>4</sub> )	0	2	2	48	44	44	49	48	44	44	49
		2	2	2	48	44	44	49	48	44	44	49
		5	2	2	48	44	44	49	50	44	44	49
		8	2	2	48	44	44	49	49	44	44	49
8	Phenol	0	2	2	42	40	40	46	46	49	56	46
		2	2	2	42	40	40	46	46	49	56	46
		5	2	2	46	40	40	46	46	49	56	46
		8	2	2	46	49	40	46	46	49	56	46
9	Hexachloro-benzene (HCB)	0	2	2	40	39	39	41	42	44	46	41
		2	2	2	40	39	39	40	41	39	43	41
		5	2	2	40	39	39	40	42	49	49	41
		8	2	2	42	43	46	40	42	39	44	41
10	Trichloro-ethene (TCE)	0	2	2	43	37	53	46	43	37	53	46
		2	2	2	43	37	53	46	43	37	53	46
		5	2	2	43	37	53	46	43	37	45	46
		8	2	2	43	37	53	46	43	37	53	46

TABLE 8. CHEMICAL ANALYSIS METHODS

Parameter of Interest	USEPA* Digestion Method	USEPA Analytical Method
Antimony	NA	7041* with Zeeman
Arsenic	NA	7760*
Barium	3020	6010†
Beryllium	3020	6010†
Cadmium	3020	7131*
Chromium	3020	7191*
Copper	3020	6010†
Lead	3020	7421*
Mercury	NA	7470*
Nickel	3020	6010†
Selenium	3020	7740* with Zeeman
Silver	3020	7761*
Thallium	3020	7841†
Zinc	3020	6010*
Volatile organics	NA	8240

\* USEPA SW-846 2nd edition (USEPA 1982).  
 USEPA SW-846 3rd edition (USEPA 1986d).

#### Preparation of Test Samples

##### WES Sludge--

Approximately 4.2 lb of Type I Portland cement was mixed with 14 lb of the 25% solids WES sludge in a Hobart C-100 mixer. A compositional analysis of the cement is presented in Table 4. After thorough mixing and prior to the initial set, this solidified/stabilized sludge was divided into two equal portions, each weighing 8.59 lb. To the first portion, 0.086 and 0.0086 lb, respectively, of each of the 12 organics listed in Table 11 was added to the cement/sludge slurry and thoroughly mixed. This resulted in cement/sludge mixtures that contained approximately 1.0% (by weight) and 0.1%, respectively, total of organics. Each of these mixtures was poured into three 1-liter

TABLE 9. BULK ANALYSIS OF WTC SOLUTION

Parameter	Ionic Species	Concentration*
Arsenic	As <sup>+3</sup>	2,400
Cadmium	Cu <sup>+2</sup>	4,600
Chromium	Cr <sup>+3</sup>	1,600
Lead	Pb <sup>+2</sup>	8,100
Phenol	--	3,700
Total solids (percent)	--	3.4
pH	--	2.5
Bulk density (g/cm <sup>3</sup> )	--	1.0

\* Expressed as milligrams per kilogram wet weight unless specified otherwise.

polyethylene bottles and sealed. The cement/sludge/organic mixtures were cured at 4° C in the sealed bottles until they were needed for testing.

#### WTC Waste---

Approximately 4.4 lb of the WTC synthetic metal solution was solidified/stabilized with 4.4 lb of Type I Portland cement, 4.4 lb of a type F flyash, and 4.4 lb of a soil. A composite analysis of the cement and flyash is given in Table 4. The soil was a Sandy Clay CL Gray Type as classified by the Unified Soil Classification system (USAEWES 1960). The waste/cement/flyash/soil mixture was split into two 8.8-lb portions. Then, 0.088 lb or 0.0088 lb of each of the organic compounds listed in Table 11 was added to each portion. The mixtures were poured into polyethylene bottles and sealed. The sealed bottles were stored at 4° C until needed for testing.

#### PCE Waste---

Unlike the WES and WTC wastes, the PCE waste was not solidified. Using the Hobart mixer, 6.6 lb of raw PCE waste was homogenized. The PCE waste was split into two 3.3-lb portions. Then, 0.033 lb or 0.0033 lb of each of the organic compounds listed in Table 11 was mixed with each portion, respectively. These mixtures were poured into polyethylene bottles and sealed. The sealed bottles were stored at 4° C until needed for testing.

#### Sample Extraction--

The WES and WTC wastes cured for a period of 14 days, and the PCE waste aged for 14 days. The waste materials were crushed in the sealed plastic bottles to minimize volatile losses. Each waste material was then ground in a

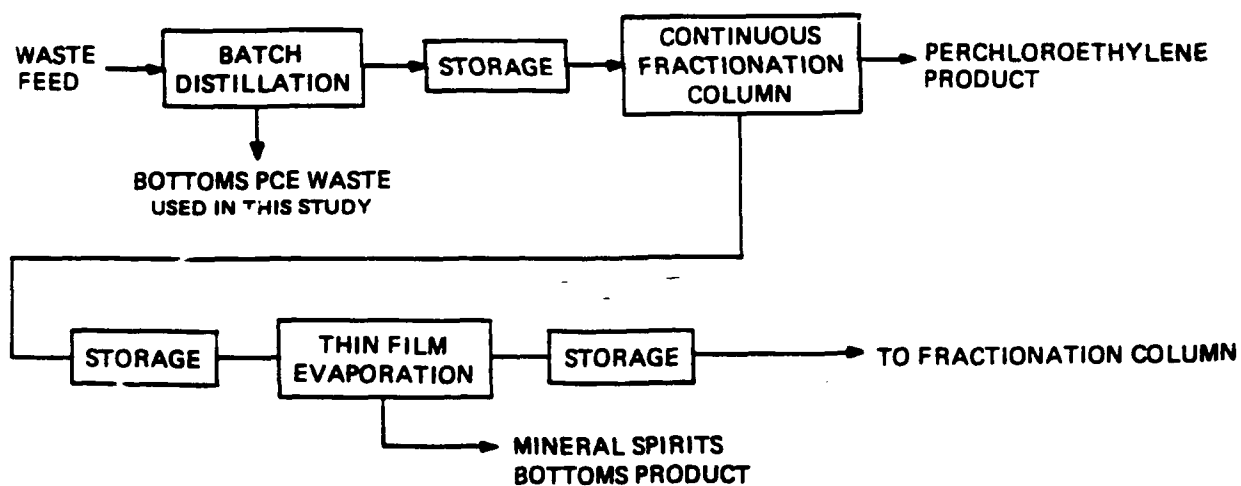


Figure 6. Flowchart of PCE waste production.

TABLE 10. BULK ANALYSES OF PERCHLOROETHYLENE WASTE

Parameter	Concentration (mg/kg)
Antimony	11.2
Barium	265
Beryllium	0.3
Cadmium	19.1
Chromium	185
Copper	2,390
Nickel	223
Silver	5.8
Zinc	1,600
Arsenic	8.9
Lead	376
Mercury	2.0
Selenium	1.7
Thallium	<1.0
Total organic halogens	4,660
Chemical oxygen demand	887,000
pH	6.07

TABLE 11. ORGANIC COMPOUNDS ADDED TO STUDY B SLUDGES

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1. Chloroform	7. 1,1,2,2-Tetrachloroethane
2. 1,2-Dichloroethane	8. Tetrachloroethene
3. 1,1,1-Trichloroethane	9. Toluene
4. Carbon Tetrachloride	10. Ethylbenzene
5. Trichloroethene	11. Methyl Ethyl Ketone
6. Benzene	12. Methyl Isobutyl Ketone

---

chilled mortar (also to minimize volatile losses) and screened through a 9.5-mm sieve. The resulting fines, for each waste, were placed in glass jars and mixed. Samples were collected from each jar for moisture analyses (Appendix C). After each waste (WES 0.1%, WES 1.0%, WTC 0.1%, WTC 1.0%, PCE 0.1%, and PCE 1.0%) was homogenized, the wastes were subjected to triplicate EP and TCLP extractions as presented in Appendices A and B. The EP was performed in tumbled, closed glass containers. The TCLP was conducted using the ZHE vessel for the extraction of volatile organics and closed glass containers for the extraction of nonvolatiles (metals).

#### Analytical Procedures

The EP and TCLP extracts were analyzed for metals and volatile organic compounds. The analytical and digestion methods used in this study are presented in Table 8. Extract samples submitted for metal analysis were digested; extract samples submitted for volatile organic analyses were not digested.

#### Spike and Recovery Study

Loss of volatile organics during conduct of the EP and the TCLP methods and subsequent sample handling was evaluated. Three volatile organic spikes, 1,1,2-trichloroethane, carbon disulfide, and chlorobenzene, were added to the extraction fluid at two points in the extraction process. Spikes were added prior to waste extraction (the prespike) and following the extraction procedure but prior to any analyses (the postspike). The volatile organic spikes chosen had a wide range of vapor pressures and solubilities. Selected properties of these volatile organic compounds are listed in Appendix D. The volatile organic compounds used as spikes were alternated as prespikes and postspikes, as listed in Table 12.

#### Quality Assurance/Quality Control

Internal and external laboratory QA/QC measures were performed for Study B. Method blanks were carried through the metal and volatile extraction every fourth sample. Duplicate, spike recovery, and surrogate recovery analyses were performed as part of internal QA/QC measures, for the volatile analyses. The method of standard addition was utilized for all metal analyses.

TABLE 12. VOLATILE SPIKE ADDITIONS FOR STUDY B

Organic Level	Extraction Test	Prespike Addition		Postspike Addition	
		Spike Compound	Concentration (mg/l)	Spike Compound	Concentration (mg/l)
<u>WES Waste</u>					
0.1%	TCLP	112 TCA*	252	CLBEN† CS2‡	120 50
	EP	--	--	112 TCA CLBEN CS2	143 55 127
1.0%	TCLP	CLBEN CS2	45 130	112 TCA	250
	EP	CLBEN CS2	50 127	112 TCA	250
<u>PCE Waste</u>					
0.1%	TCLP	112 TCA	82	CLBEN CS2	20 48
	EP	--	--	CLBEN CS2	21 20
1.0%	TCLP	CLBEN CS2	20 48	--	--
	EP	CLBEN CS2	20 30	--	--
<u>WTC Waste</u>					
0.1%	TCLP	112 TCA CS2	15 25	CLBEN	7
	EP	112 TCA CS2	15 25	CLBEN	7
1.0%	TCLP	CS2	30	CLBEN	10
	EP	CS2	30	CLBEN	10

\* 1,1,2-Trichloroethane.

† Chlorobenzene.

‡ Carbon disulfide.

## STATISTICAL PROCEDURES

Statistical analyses were performed, using the Statistical Analysis System (SAS) software package provided by SAS Institute, Inc. (1987). An analysis of the variance multifactor factorial test, as described by Miller and Freund (1985), was conducted on data sets produced by Study A and Study B. An analysis of variance (ANOVA) procedure outlined in Chapter 11 of the SAS/STAT user guide (SAS Institute, Inc. 1987) was used to perform this statistical procedure.

When it was determined that the levels of interaction were significant, a "paired-sample T test" (Miller and Freund 1985) was used to determine if the EP and TCLP results differed significantly. A MEANS procedure outlined in Chapter 33 of the SAS/STAT user guide (SAS Institute, Inc. 1987) was used to perform this statistical procedure.

Concentrations below detection levels were estimated by dividing the detection level by 2 rather than using the actual detection level or zero, as an estimate of the concentration. This is an accepted method of reporting concentration values near the detection limit (Francis and Maskarinec 1986).

The multifactor factorial experimental designs for Study A and Study B are illustrated in Tables 13 and 14, respectively. One multifactor factorial method was performed for each contaminant. Decisions on whether to reject or accept the null hypothesis were made using an alpha level of significance of 0.05, or 20:1 odds.

TABLE 13. STUDY A MULTIFACTOR FACTORIAL EXPERIMENTAL DESIGN

Level	A. Interference Compound	B. Interference Concentration	C. Extraction Test	D. Replicate
1	Oil	0%	TCLP	1
2	Grease	2%	EP	2
3	HCb*	5%		
4	Phenol	8%		
5	TCE†			
6	Lead nitrate			
7	Zinc nitrate			
8	Copper nitrate			
9	Sodium hydroxide			
10	Sodium sulfate			

\* Hexachlorobenzene.

† Trichloroethene.

TABLE 14. STUDY B MULTIFACTOR FACTORIAL EXPERIMENTAL DESIGN

Level	A. Sludge Type	B. Organic Concentration	C. Extrac- tion Test	D. Replicate
1	WES	1.0%	TCLP	1
2	WTC	0.1%	EP	2
3	PCE			

## SECTION 5

### RESULTS AND DISCUSSION

#### STUDY A

The results from the EP and TCLP extractions conducted during Study A are presented in Tables 15 and 16 and Figures 7 through 10. Raw data for each sample subjected to an EP or TCLP extraction are presented in Appendix E.

Table 15 presents the average (averaged over the duplicate samples) extract concentrations for the TCLP and EP test for each contaminant. Summary statistics for this data set are presented in Table 16. The values presented in Table 16 are averaged across the different interference compounds and concentrations and thus cannot be utilized for a detailed interpretation of the data. However, this information can be used to visualize general trends in the data set. Table 16 indicates that a larger concentration of mercury is detected in the TCLP and EP leachates than the other metals. Table 16 also indicates that the TCLP average extract values for chromium are 1.3 times larger than the average EP extract values.

To establish a basis for comparing the many batches of sludge that were extracted as part of Study A, it was necessary to normalize the data. The extract concentrations that were compared in this study were normalized to their dry-raw waste concentration. Normalization corrects for dilution by the interference materials, small changes in the binder ratio, and variations in the moisture contents of the extracted materials. Normalized extract concentrations were derived using the following equation:

$$EC_n = (EC * V) / (W * M * B) \quad (1)$$

where  $EC_n$  = normalized extract concentration, mg/kg

EC = contaminant concentration measured in the TCLP or EP extract, mg/l

V = volume of extraction fluid, liters

W = weight of the wet treated waste extracted, kg

M = solids concentration of the solidified/stabilized waste extracted, expressed as a decimal

B = weight fraction of raw waste in the solidified/stabilized/interfered waste mixture, calculated as follows:

$$\frac{\text{weight of raw waste}}{(\text{weight of raw waste} + \text{weight of binder} + \text{weight of interference agent})} \quad (2)$$

Results of the analysis of the variance multifactor factorial test (AVMFT) performed on the Study A normalized extract concentrations are presented in Table 17. When the results of the AVMFT indicated the levels of interactions between the tests and the other variables were significant, a paired-sample T test was also performed. If the test interactions are significant, the paired T test result must be utilized to evaluate the data. The

TABLE 15. STUDY A: AVERAGE TCLP AND EP EXTRACT CONCENTRATIONS

Interference Compound	Interference Concentration	Average Extract Concentration (mg/l)					
		Cadmium		Chromium		Nickel	
		EP	TCLP	EP	TCLP	EP	TCLP
Oil	0%	0.0207	0.0044	0.400	0.092	0.069	0.049
	2%	0.00335	0.0015	0.017	0.0605	0.0645	0.0375
	5%	0.00175	0.014	0.020	0.0185	0.0655	0.0125
	8%	0.0036	0.00795	0.017	0.045	0.0645	0.0725
Grease	0%	0.0086	0.00045	0.2465	0.044	0.1775	0.0265
	2%	0.0104	0.0001	0.052	0.011	0.017	0.0145
	5%	0.0058	0.0002	0.048	0.0285	0.006	0.0115
	8%	0.01035	0.0001	0.0145	0.033	0.0085	0.0085
Lead	0%	0.002	0.00165	0.049	0.0505	0.0125	0.031
	2%	0.00465	0.0095	0.0365	0.0565	0.0085	0.015
	5%	0.0053	0.0925	0.030	0.0435	0.0075	0.0475
	8%	0.04395	0.0224	0.030	0.0615	0.025	0.0685
Copper	0%	0.00275	0.0001	0.0095	0.0385	0.017	0.0215
	2%	0.00275	0.00055	0.0875	0.068	0.0235	0.0285
	5%	0.0033	0.0001	0.036	0.0485	0.0145	0.028
	8%	0.0016	0.00045	0.0105	0.046	0.0175	0.0515
Zinc	0%	0.04785	0.00555	0.035	0.068	0.065	0.033
	2%	0.0079	0.00145	0.0455	0.062	0.091	0.0033
	5%	0.00615	0.00435	0.084	0.098	0.092	0.011
	8%	0.00345	0.00235	0.0955	0.094	0.1035	0.0685
Hexachloro- benzene	0%	0.02265	0.00275	0.028	0.300	0.0195	0.088
	2%	0.0066	0.00215	0.0505	0.240	0.0145	0.059
	5%	0.0217	0.01275	0.0105	0.5105	0.014	0.1515
	8%	0.0054	0.0002	0.0605	0.035	0.1475	0.024

(Continued)

TABLE 15. (Concluded)

Interference Compound	Interference Concentration	Average Extract Concentration (mg/l)							
		Cadmium		Chromium		Nickel		Mercury	
		EP	TCLP	EP	TCLP	EP	TCLP	EP	TCLP
Trichloro- ethene	0%	0.00145	0.00035	0.0405	0.0765	0.0145	0.0045	0.422	0.228
	2%	0.0014	0.0001	0.047	0.0825	0.0095	0.0075	0.380	0.277
	5%	0.00135	0.0001	0.0365	0.0725	0.011	0.0115	0.622	0.424
	8%	0.0007	0.0001	0.0505	0.065	0.010	0.004	0.395	0.670
Sodium sulfate	0%	0.00645	0.0013	0.071	0.049	0.0615	0.0945	0.170	0.252
	2%	0.0074	0.0009	0.126	0.1545	0.058	0.070	0.145	0.281
	5%	0.00675	0.00095	0.153	0.1435	0.065	0.063	0.115	0.206
	8%	0.00845	0.002	0.153	0.145	0.0625	0.0555	0.156	0.205
Sodium hydroxide	0%	0.0006	0.00025	0.085	0.080	0.0795	0.065	0.146	0.153
	2%	0.0053	0.0003	0.132	0.1155	0.0595	0.055	0.277	0.197
	5%	0.00305	0.0001	0.484	0.413	0.007	0.067	0.310	0.190
	8%	0.002	0.0001	0.379	0.317	0.002	0.0365	0.289	0.286
Phenol	0%	0.0021	0.00415	0.0145	0.098	0.009	0.076	0.332	0.369
	2%	0.0030	0.0001	0.0085	0.162	0.005	0.079	0.310	1.30
	5%	0.00465	0.0001	0.0055	0.0475	0.0075	0.036	1.30	1.33
	8%	0.0015	0.002	0.0195	0.0155	0.0045	0.0395	1.32	1.42

TABLE 16. SUMMARY STATISTICS FOR STUDY A METALS DATA

Statistic	Metal Contaminant (mg/l)							
	Cd		Cr		Ni		Hg	
	EP	TCLP	EP	TCLP	EP	TCLP	EP	TCLP
Number of samples	80	80	80	80	80	80	80	80
Maximum value	0.096	0.096	0.485	0.689	0.28	0.203	1.32	1.48
Minimum value	<0.001	<0.001	0.004	0.007	<0.001	<0.001	0.001	0.002
Average value	0.008	0.005	0.075	0.104	0.04	0.044	0.312	0.347
Standard deviation	0.014	0.005	0.096	0.113	0.047	0.036	0.313	0.315
Range	0.095	0.095	0.481	0.682	0.279	0.202	1.32	1.48
Coefficient of variation	175	100	128	109	118	81.8	100	90.8
Detection limit*	<0.0001	<0.0001	<0.001	<0.001	<0.001	<0.001	<0.0002	<0.0002

\* The detection limit for the reported contaminant.

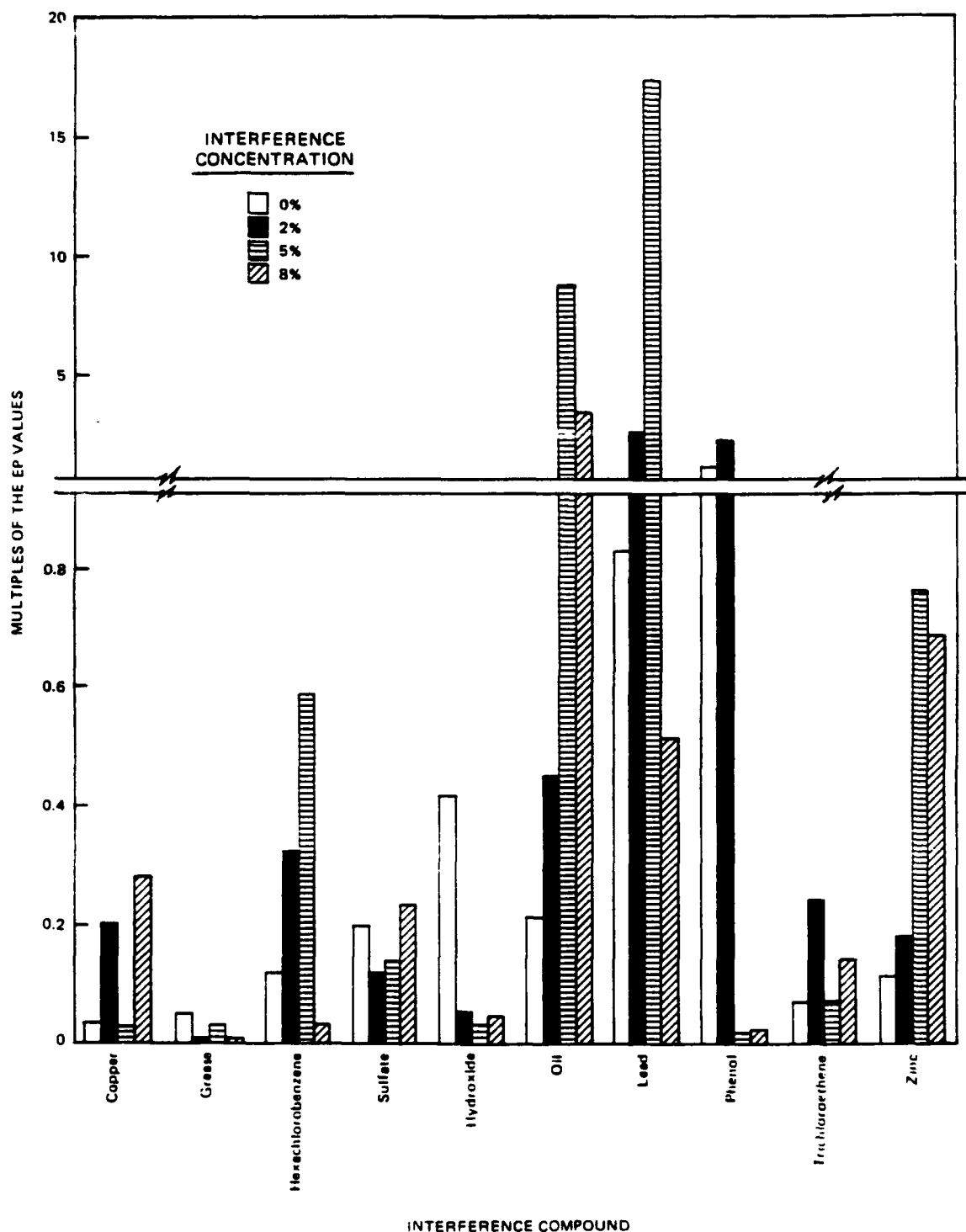


Figure 7. Average normalized Study A cadmium extract concentrations expressed as the TCLP concentration divided by the EP concentration.

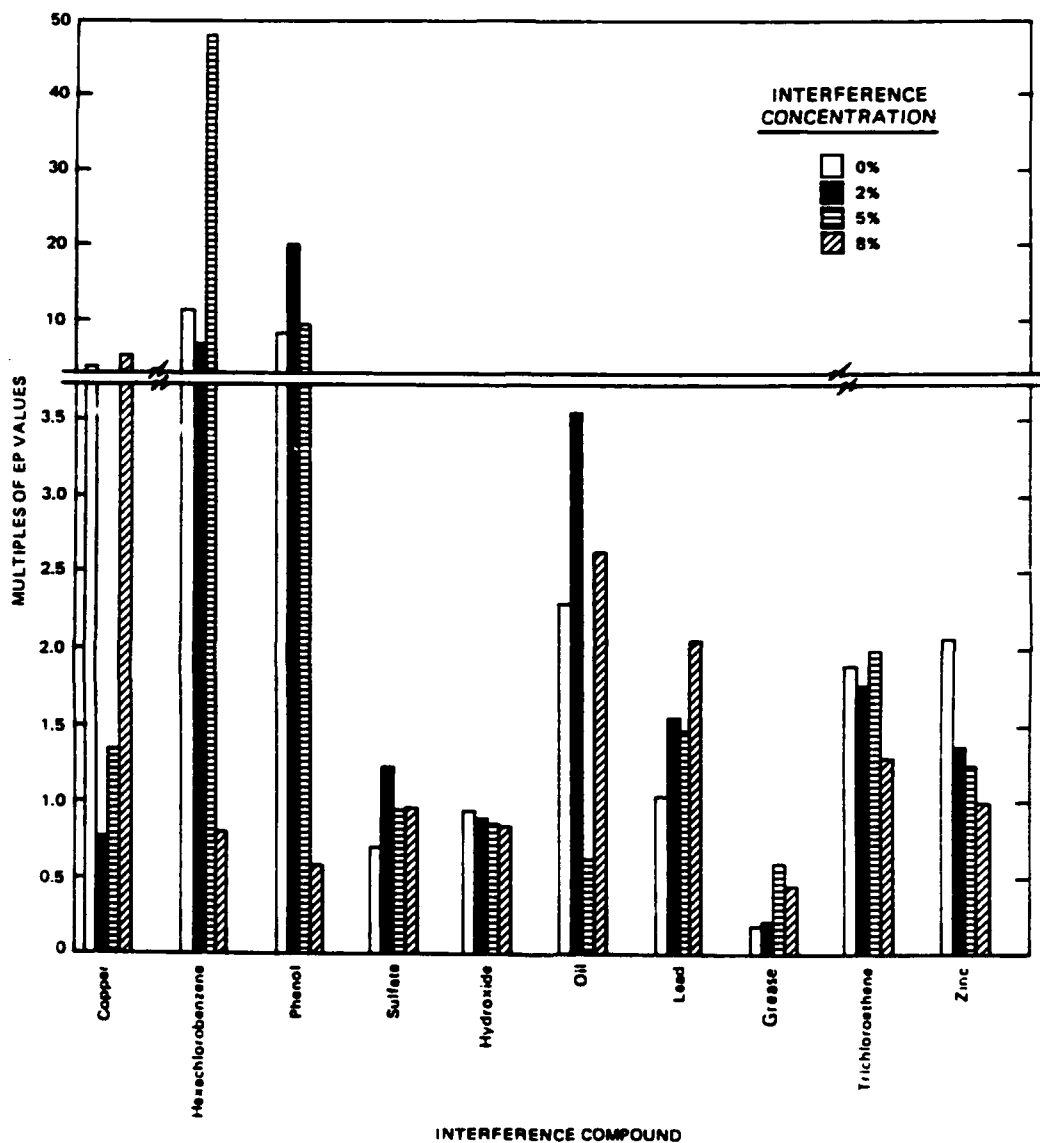


Figure 8. Average normalized Study A chromium extract concentrations expressed as the TCLP concentration divided by the EP concentration.

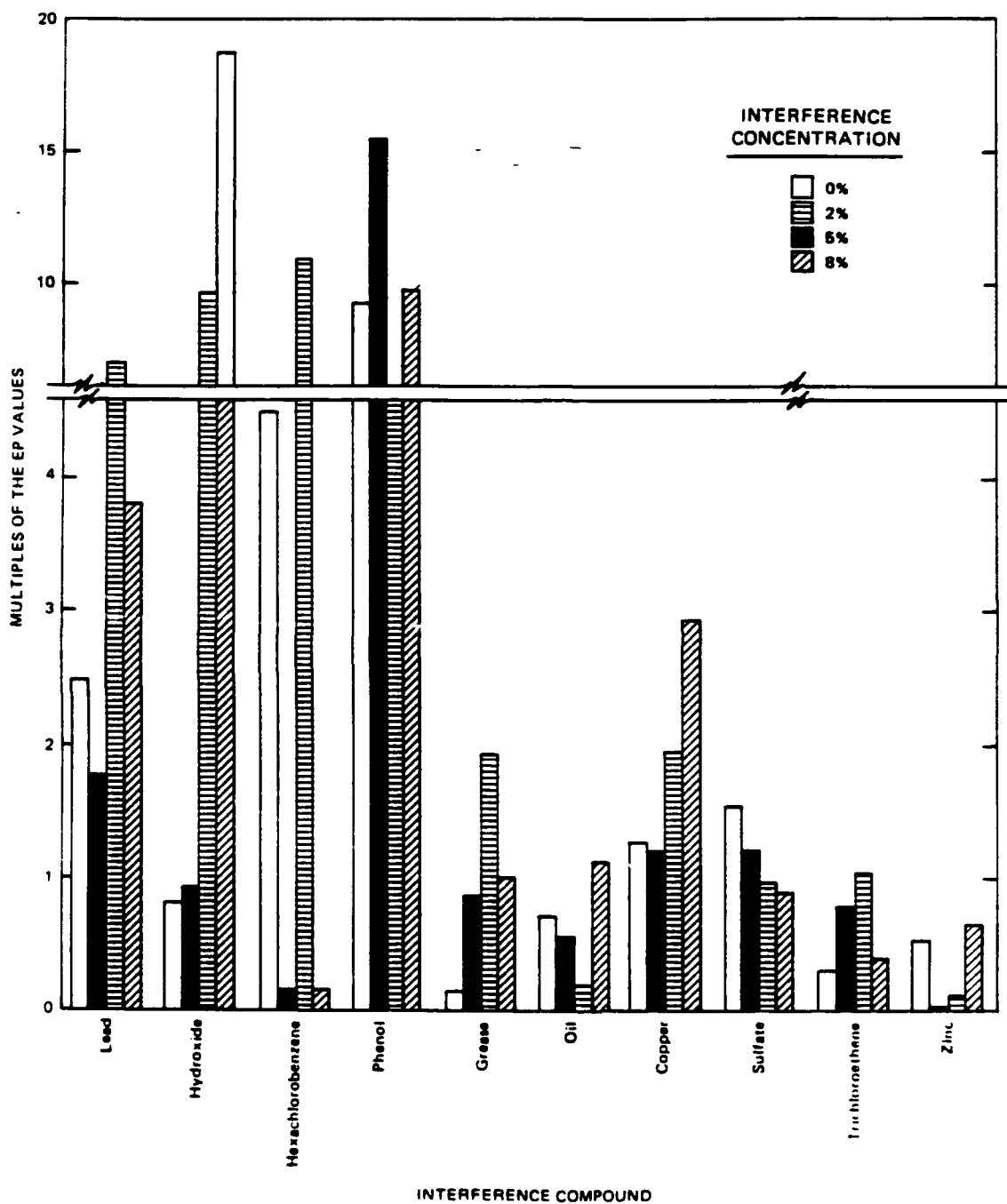


Figure 9. Average normalized Study A nickel concentrations expressed as the TCLP concentration divided by the EP concentration.

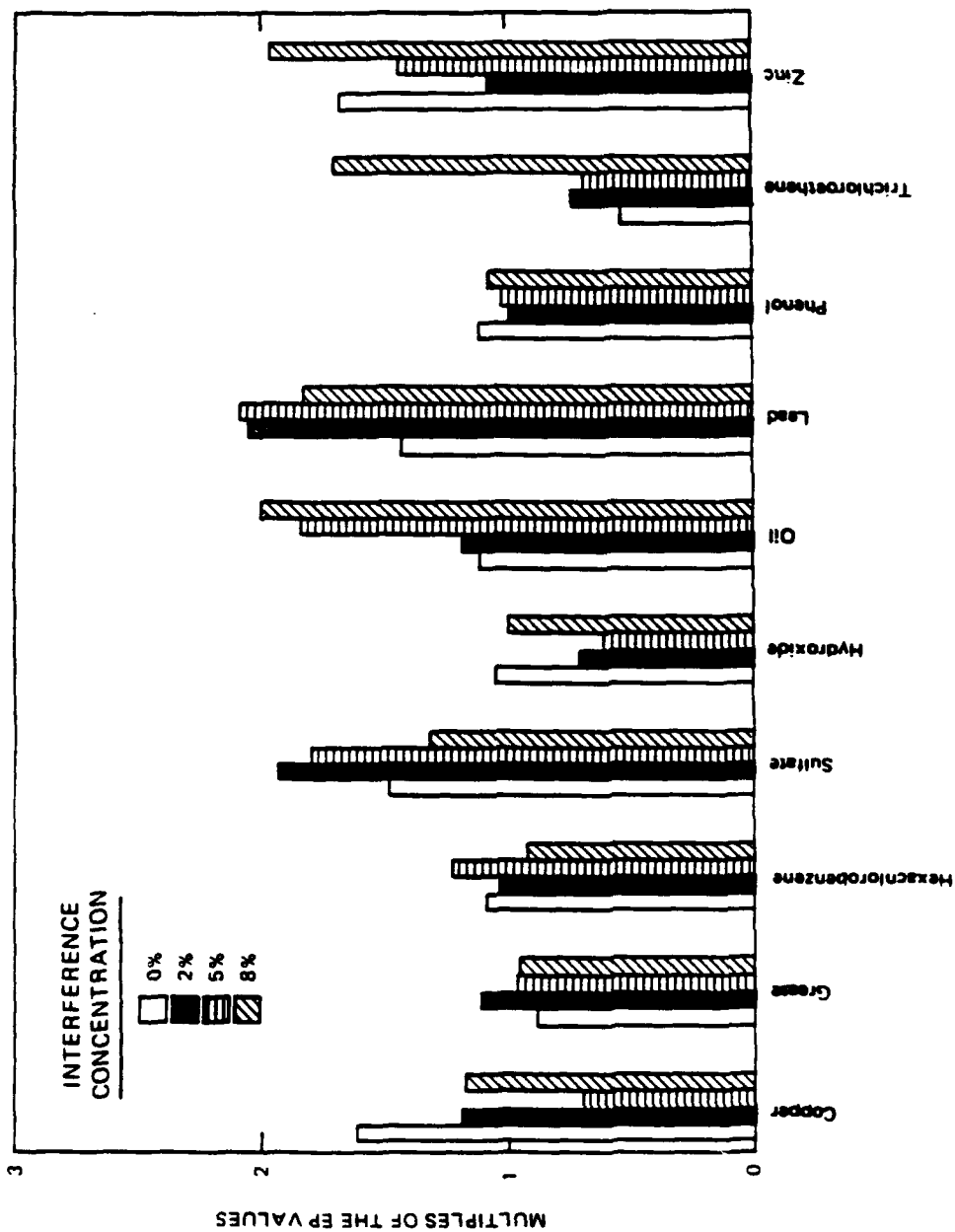


Figure 10. Average normalized Study A mercury extract concentrations expressed as the TCLP concentration divided by the EP concentration.

TABLE 17. RESULTS OF AVMFT PERFORMED ON NORMALIZED STUDY A TCLP  
AND EP METALS RESULTS

Metal Contaminant	Inter- ference Compound*	Interference Concentration†	Extrac- tion Test‡	Replicate	Test Inter- action	R Value§
Cadmium	Y	N	N	N	N	0.5961
Chromium	Y	Y	Y	N	N	0.9413
Nickel	Y	N	N	N	Y	0.7716
Mercury	Y	Y	Y	N	Y	0.9826

(Results Presented as "Yes or No")#

\* Compounds are listed in Table 6.

† Interference concentrations = 0%, 2%, 5%, and 8%.

‡ EP and TCLP.

§ R values give an indication of how well the statistical model fits the data. As the fit of the model improves, the R value approaches 1.0.

# Yes (Y) indicates there is statistical difference between the variables compared at  $\alpha = 0.05$ .

TABLE 18. RESULTS OF PAIRED-SAMPLE T TEST PER-  
FORMED ON NORMALIZED STUDY A TCLP AND EP  
NICKEL AND MERCURY DATA

Metal Contaminant	Extraction Test*
Nickel	N
Mercury	Y

Note: The paired T test was performed only when the levels of interaction were found to be significant in the AVMFT.

\* Yes (Y) indicates there is statistical difference between the EP and TCLP results at  $\alpha = 0.05$ .

results of the paired T test are presented in Table 18. As indicated by Tables 17 and 18, using a level of significance of  $\alpha = 0.05$ , the results of the TCLP and EP extractions for chromium and mercury are statistically different, while the results for nickel and cadmium contaminants were not statistically different.

Results of the TCLP and EP extractions for each metal contaminant in Study A are presented in Appendix F, Figures F1 through F4. In these figures, the normalized EP extract concentrations are plotted versus the normalized TCLP extract concentrations. A discussion accompanies these figures.

Figures 7 through 10 present the normalized TCLP and EP extracts expressed as multiples of the average EP values for the duplicate samples. The values presented in these figures were calculated as shown by the following equation:

$$\frac{(\text{TCLP}_1 + \text{TCLP}_2)/2}{(\text{EP}_1 + \text{EP}_2)/2} \quad (3)$$

where  $\text{TCLP}_1$  and  $\text{TCLP}_2$  = normalized TCLP replicate extract concentration for the contaminant of interest, mg/g

$\text{EP}_1$  and  $\text{EP}_2$  = normalized EP replicate extract concentration for the contaminant of interest, mg/g

Thus, for these figures, a value of 1.0 indicates that the amount of a particular contaminant measured in the TCLP extract is equal to the amount of that contaminant measured in the EP extracts. Values greater than 1.0 indicate that the TCLP extract concentration is greater than the EP extract concentration, and values less than 1.0 indicate that the EP concentration is greater than the TCLP.

Figure 9, showing the nickel data, indicates that for the majority of the conditions evaluated, the EP and TCLP produce similar results. Figures 8 and 10 illustrate that the TCLP extraction is more aggressive for chromium and mercury. Figure 10 (the mercury data) indicates that of the 40 conditions investigated in Study A, 28 resulted in TCLP extracts containing higher concentrations of mercury. Figure 8 (the chromium data) indicates that 25 of the 40 conditions resulted in TCLP extracts containing higher concentrations of chromium.

It is interesting to note that inspection of Figure 7 provides information which is in direct conflict with the results of the statistical analysis. Figure 7 (the cadmium data) indicates that, for 33 of the 40 conditions evaluated, the EP extracts contained higher concentrations of cadmium. Figure 7 indicates that the results of the EP and TCLP differ, while the statistical results presented in Table 17 indicate no difference between the extract concentrations. Based on this information, there is a possibility that a Type II error was made. (A Type II error occurs when the results of the EP and TCLP extraction are actually different but this is not revealed by the analysis of the variance statistic.)

Although it is interesting that for some contaminants the EP and TCLP extraction results differ, it is beyond the scope of this study to pinpoint the variables that are responsible for the dissimilarities. However, there is one observation that should be noted. Due to the fact that every TCLP extraction for Study A utilized extraction fluid 2, and every EP extraction required the full 400 ml of 0.5 acetic acid (Appendix A), the buffering capacity of the EP and TCLP extraction fluids was equal. This leads to the conjecture that the EP and TCLP extractions should be similar in their aggressiveness. Contrary to the similarity between extraction fluids, the TCLP results varied from the EP results for mercury and chromium. Consequently, the variations between the EP and TCLP extracts cannot be attributed just to pH influences but must be a function of other differences between the extraction procedures, such as time of extraction, method of agitation, etc.

## STUDY B

### Results for the Metal Contaminants

The results for the Study B metal EP and TCLP extraction tests are presented in Tables 19 through 22 and Figure 11. Raw data for each sample subjected to the EP or TCLP extraction for metal compounds are presented in Appendix G, Tables G1 through G3. Table 19 presents the average (averaged over the three replicates) metal extract concentrations for the TCLP and EP tests. Results presented in this table generally indicate that the TCLP-generated extracts contained higher concentrations of the metal contaminants than the EP extracts.

Summary statistics for this data set are presented in Table 20. As indicated in this table, 10 of the 15 average metal values were higher in the TCLP than the EP extracts. This table also illustrates that the EP data generally varied over a larger range than the TCLP data.

Results of the AVMFT performed on the Study B metal data are presented in Table 21. As in Study A, when the results of the AVMFT indicate that the levels of test interaction are significant, a paired T test was performed. Results of the paired T test are presented in Table 22. Statistical analysis for the WES waste indicates that there is not a significant difference between the EP and TCLP extraction for any of the metals except mercury. The statistical analysis for the WTC waste indicates that the EP and TCLP differ significantly for arsenic and lead and were not statistically different for chromium. The results of the PCE waste extractions indicated that there were statistical differences between concentrations of copper, zinc, and barium contaminants measured in the TCLP and EP extracts. Several values are reported in Table 21 as "DL." This indicates that the concentration of these contaminants were, in the TCLP and EP extracts, at or below the detection limits. These extracts have no basis for comparison; consequently, the results for the PCE-arsenic, PCE-silver, and WTC-cadmium are omitted for the remainder of the discussion.

A graphical representation of the results of the TCLP and EP extractions for each metal contaminant in Study B is presented in Appendix H, Figures H1 through H7. In these figures, the normalized EP extract concentrations are plotted versus the normalized TCLP extract concentrations. A discussion of the results accompanies these figures.

TABLE 19. STUDY B AVERAGE TCLP AND EP EXTRACT CONCENTRATIONS FOR  
METAL CONTAMINANTS (AVERAGED OVER THREE REPLICATE SAMPLES)

Metal Contaminant	Sludge	Organic Level Percentage	Extract Concentration (mg/l)	
			EP	TCLP
Antimony	WES	0.1	NA*	NA
		1.0	NA	NA
	WTC	0.1	NA	NA
		1.0	NA	NA
	PCE	0.1	0.0273	0.0367
		1.0	0.023	0.038
Arsenic	WES	0.1	NA	NA
		1.0	NA	NA
	WTC	0.1	0.021	0.055
		1.0	0.028	0.121
	PCE	0.1	0.0041	<0.005
		1.0	0.005	0.007
Barium	WES	0.1	NA	NA
		1.0	NA	NA
	WTC	0.1	NA	NA
		1.0	NA	NA
	PCE	0.1	0.382	0.459
		1.0	0.334	0.561
Cadmium	WES	0.1	0.001	0.010
		1.0	0.030	0.007
	WTC	0.1	0.0004	0.0002
		1.0	<0.0001	0.0018
	PCE	0.1	NA	NA
		1.0	NA	NA
Chromium	WES	0.1	0.024	0.070
		1.0	0.130	0.056
	WTC	0.1	0.041	0.040
		1.0	0.032	0.036
	PCE	0.1	NA	NA
		1.0	NA	NA
Copper	WES	0.1	NA	NA
		1.0	NA	NA
	WTC	0.1	NA	NA
		1.0	NA	NA
	PCE	0.1	10.747	13.067
		1.0	10.833	16.333

(Continued)

\* Not analyzed.

TABLE 19. (Concluded)

Metal Contaminant	Sludge	Organic Level Percentage	Extract Concentration (mg/l)	
			EP	TCLP
Lead	WES	0.1	NA	NA
		1.0	NA	NA
	WTC	0.1	0.007	0.228
		1.0	0.013	0.044
	PCE	0.1	0.036	0.065
		1.0	0.028	0.074
Mercury	WES	0.1	7.957	7.843
		1.0	0.019	8.310
	WTC	0.1	NA	NA
		1.0	NA	NA
	PCE	0.1	NA	NA
		1.0	NA	NA
Nickel	WES	0.1	0.021	0.120
		1.0	0.184	0.205
	WTC	0.1	NA	NA
		1.0	NA	NA
	PCE	0.1	NA	NA
		1.0	NA	NA
Silver	WES	0.1	NA	NA
		1.0	NA	NA
	WTC	0.1	NA	NA
		1.0	NA	NA
	PCE	0.1	0.002	<0.001
		1.0	0.004	<0.001
Zinc	WES	0.1	NA	NA
		1.0	NA	NA
	WTC	0.1	NA	NA
		1.0	NA	NA
	PCE	0.1	29.267	32.200
		1.0	16.733	32.933

TABLE 20. SUMMARY STATISTICS FOR STUDY B METALS DATA

Sludge	Contaminant	Maximum		Minimum		Average		Detection Limit (mg/l)
		Value (mg/l)		Value (mg/l)		Value (mg/l)		
		EP	TCLP	EP	TCLP	EP	TCLP	
WES	Cadmium	0.06	0.02	0.0006	0.0051	0.016	0.009	<0.0001
	Chromium	0.31	0.10	0.019	0.048	0.077	0.063	<0.001
	Nickel	0.35	0.24	0.011	0.096	0.103	0.163	<0.001
	Mercury	8.48	8.56	0.017	7.65	3.99	8.08	<0.00008
WTC	Arsenic	0.03	0.14	0.02	0.05	0.024	0.088	<0.005
	Cadmium	0.0006	0.0005	0.0001	0.0001	0.0003	0.0002	<0.0001
	Chromium	0.04	0.05	0.03	0.04	0.037	0.136	<0.001
	Lead	0.02	0.32	0.005	0.04	0.010	0.038	<0.001
PCE	Antimony	0.03	0.04	0.02	0.03	0.03	0.04	<0.005
	Arsenic	0.007	0.008	0.005	0.006	0.005	0.007	<0.005
	Copper	13.1	16.5	9.30	12.9	10.79	14.7	<0.0001
	Lead	0.05	0.085	0.02	0.0003	0.03	0.032	<0.001
	Silver	0.004	0.009	0.001	0.001	0.003	0.002	<0.001
	Zinc	36.3	33.4	16.4	31.6	23.0	32.6	<0.003
	Barium	0.44	0.58	0.315	0.43	0.358	0.51	<0.001

(Continued)

TABLE 20. (Concluded)

Sludge	Contaminant	Standard Deviation		Range (mg/l)		Coefficient of Variation		Number of Samples
		EP	TCLP	EP	TCLP	EP	TCLP	
WES	Cadmium	0.022	0.004	0.059	0.015	137	44.4	6
	Chromium	0.106	0.017	0.291	0.052	137	27.0	6
	Nickel	0.124	0.050	0.339	0.144	120	30.7	6
	Mercury	3.98	0.307	8.46	0.910	99.7	3.78	6
WTC	Arsenic	0.004	0.034	0.100	0.090	16.7	38.6	6
	Cadmium	0.0002	0.0002	0.0005	0.0004	66.7	100.0	6
	Chromium	0.005	0.005	0.100	0.010	13.5	3.66	6
	Lead	0.003	0.103	0.015	0.280	30.0	271	6
PCE	Antimony	0.003	0.002	0.010	0.010	10.0	5.00	6
	Arsenic	0.0007	0.0007	0.002	0.002	14.0	10.0	6
	Copper	1.19	1.64	3.80	3.60	11.79	11.2	6
	Lead	0.010	0.008	0.030	0.085	33.3	25.0	6
	Silver	0.001	0.003	0.003	0.008	33.3	150.0	6
	Zinc	7.32	0.576	19.9	1.80	31.8	1.77	6
	Barium	0.046	0.059	0.125	0.150	12.8	11.6	6

TABLE 21. RESULTS OF STATISTICAL ANALYSIS FOR NORMALIZED  
STUDY B TCLP AND EP METAL EXTRACTS

Sludge	Metal Contaminant	Organic Levels*	Extraction Test†	Replicate	Test Inter-action	R Value‡
WES	Cadmium	N	N	N	N	0.598
	Chromium	N	N	N	N	0.424
	Nickel	N	N	N	N	0.653
	Mercury	Y	Y	N	Y	0.973
WTC	Arsenic	Y	Y	N	Y	0.973
	Cadmium	DL	DL	DL	--	--
	Chromium	N	N	N	N	0.530
	Lead	Y	Y	N	Y	0.906
PCE	Antimony	N	Y	N	N	0.947
	Arsenic	DL	DL	DL	--	--
	Copper	Y	Y	N	Y	0.929
	Lead	N	Y	N	N	0.878
	Silver	DL	DL	DL	--	--
	Zinc	N	Y	N	Y	0.902
	Barium	N	Y	N	Y	0.913

Note: Results presented as "Yes (Y)" or "No (N)." Yes indicates there is statistical difference between the variable compared at  $\alpha = 0.05$ .  
DL = detection limit.

\* 0.1% and 1.0%.

† EP and TCLP.

‡ R values give an indication of how well the statistical model fits the data. As the fit of the model improves, the R value approaches 1.0.

Figure 11 presents, for all three sludges, the normalized TCLP and EP extracts expressed as multiples of EP values averaged for the replicate samples. The figure illustrates that the TCLP is a more aggressive extraction for the metal contaminants. On the average, the extract from the TCLP contained concentrations of metals approximately twice as large as the metal concentrations measured in EP extracts.

The results of the Study B metal extractions are summarized as follows.

(1) The results of the statistical analysis indicate that, for the PCE waste, the TCLP and EP extractions produce extracts that are significantly different. This may be explained by the fact that the PCE sludge had a pH of 6 and was not solidified/stabilized. Because of the low alkalinity of this material, extraction fluid 1 was used for the TCLP extraction, and little acid was added in the EP extraction. Thus, the TCLP and EP extraction fluids were substantially different. It is suspected that the results of the TCLP and EP extractions varied as the result of the difference in extraction fluids.

TABLE 22. RESULTS OF PAIRED-SAMPLE T TEST FOR NORMALIZED  
STUDY B TCLP AND EP METAL EXTRACTS

Sludge	Metal Contaminant	Extraction Test*
WES	Cadmium	--
	Chromium	--
	Nickel	--
	Mercury	N
WTC	Arsenic	Y
	Cadmium	--
	Chromium	--
	Lead	Y
PCE	Antimony	--
	Arsenic	--
	Copper	Y
	Lead	--
	Silver	--
	Zinc	Y
	Barium	Y

Note: The paired T test was performed only when the levels of interaction were found to be significant in the AVMFT.

\* Yes (Y) indicates there is statistical difference between the EP and TCLP results at  $\alpha = 0.05$ .

(2) For a majority of the cases studied, the WES and WTC wastes produced TCLP and EP extracts that were not statistically different. Arsenic and lead were the only contaminants for which the TCLP and EP statistically differed. One possible explanation for the EP and TCLP generating extracts with similar contaminant concentrations is that the WTC and WES wastes were solidified/stabilized, resulting in high alkalinity. Consequently, the TCLP extraction for the WES and WTC wastes required the use of extraction fluid 2. The EP extraction, performed on the WES and WTC wastes, also required the addition of the full 400 ml of acetic acid because of the low alkalinity. When 400 ml of 0.5 N acetic acid is added to 1,600 ml of water, the alkaline neutralization capacity of the EP extraction fluid and the TCLP's extraction fluid 2 are equal. Equal alkaline neutralization capacity offers one explanation for the WTC and WES sludges producing similar TCLP and EP extracts.

#### Results for the Organic Contaminants

The results of the organic analyses for the Study B extraction procedures are presented in Tables 23 through 25 and Figures 12 through 13. The raw data for each sample subjected to an EP or TCLP extraction for the organic compounds are also presented in Appendix I, Tables I1 through I12. Table 23 presents the average (averaged over the three replicates) extract concentrations for the TCLP and EP tests. The results presented in this table indicate

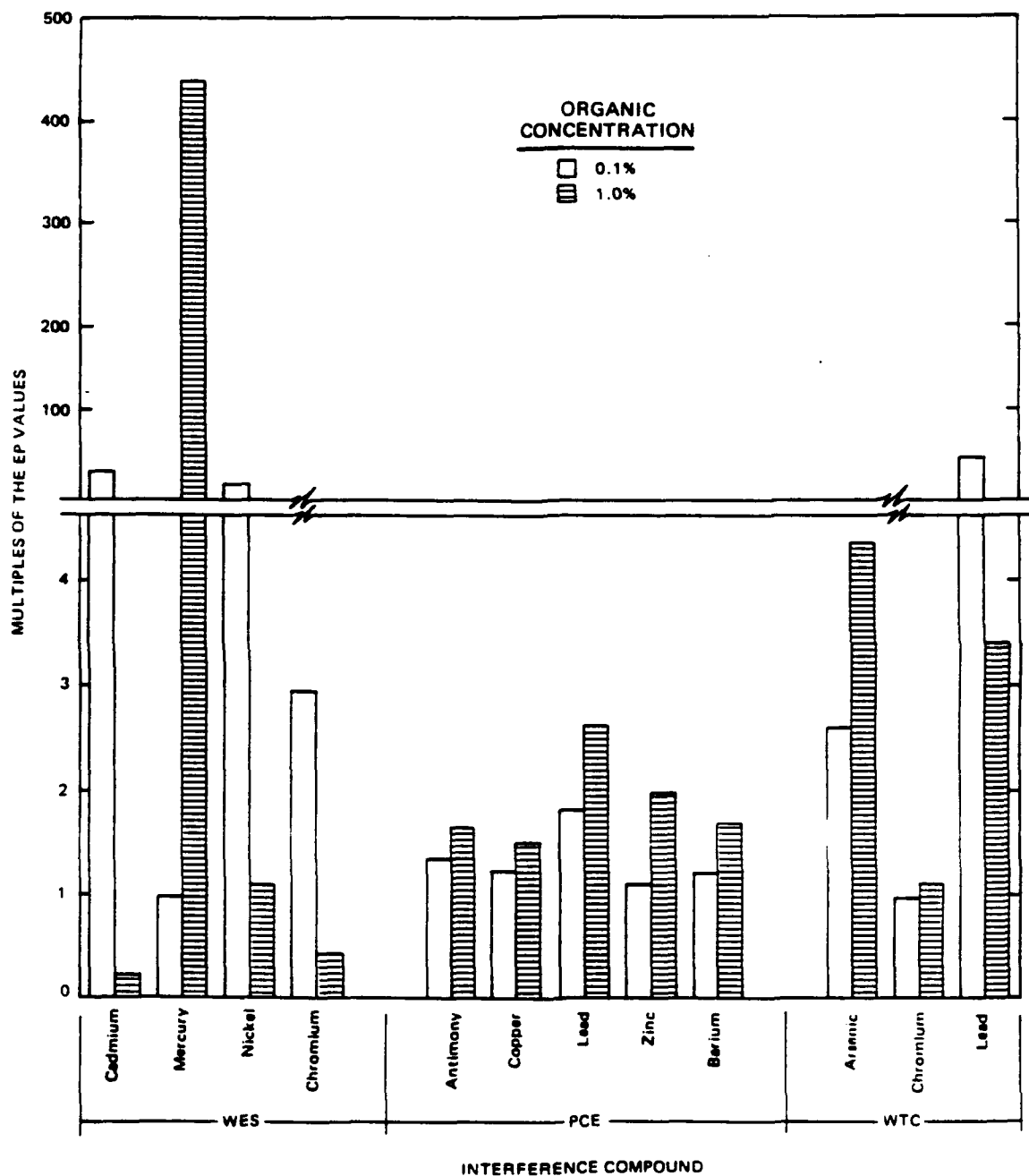


Figure 11. Average normalized Study B metal extract concentrations expressed as the TCLP concentration divided by the EP concentration.

TABLE 23. STUDY B AVERAGE TCLP AND EP EXTRACT CONCENTRATIONS  
FOR THE ORGANIC CONTAMINANTS  
(AVERAGED OVER THREE REPLICATE EXTRACT SAMPLES)

Organic Contaminant	Sludge	Organic Level	Extract Concentration (mg/l)	
			EP	TCLP
<u>Chloroform</u>	WES	0.1%	0.88	1.40
		1.0%	13.97	27.27
	PCE	0.1%	1.01	1.56
		1.0%	23.77	32.70
	WTC	0.1%	0.22	.20
		1.0%	8.98	9.13
<u>1,2-Dichloroethane</u>	WES	0.1%	1.57	1.27
		1.0%	38.70	61.37
	PCE	0.1%	3.61	4.23
		1.0%	57.30	71.40
	WTC	0.1%	0.76	0.49
		1.0%	45.03	44.23
<u>1,1,1-Trichloroethane</u>	WES	0.1%	0.96	1.93
		1.0%	18.33	46.80
	PCE	0.1%	0.55	4.80
		1.0%	15.07	25.07
	WTC	0.1%	0.29	0.45
		1.0%	15.07	24.83
<u>Carbon Tetrachloride</u>	WES	0.1%	0.42	0.89
		1.0%	3.93	7.60
	PCE	0.1%	0.23	0.50
		1.0%	10.00	10.00
	WTC	0.1%	0.10	0.20
		1.0%	5.00	5.00
<u>Trichloroethene</u>	WES	0.1%	3.47	6.90
		1.0%	64.63	134.33
	PCE	0.1%	1.48	3.54
		1.0%	33.73	39.97
	WTC	0.1%	2.32	2.55
		1.0%	98.07	135.67

(Continued)

(Sheet 1 of 2)

TABLE 23. (Continued)

Organic Contaminant	Sludge	Organic Level	Extract Concentration (mg/l)	
			EP	TCLP
<u>Benzene</u>	WES	0.1%	1.60	2.30
		1.0%	42.97	85.33
	PCE	0.1%	2.62	5.29
		1.0%	54.17	76.57
	WTC	0.1%	0.91	0.79
		1.0%	55.23	62.40
<u>1,1,2,2-Tetrachloroethane</u>	WES	0.1%	0.25	0.22
		1.0%	1.00	5.00
	PCE	0.1%	7.31	9.04
		1.0%	92.70	79.63
	WTC	0.1%	0.10	0.20
		1.0%	5.00	5.00
<u>Tetrachloroethene</u>	WES	0.1%	3.10	7.00
		1.0%	25.97	38.67
	PCE	0.1%	3.03	3.19
		1.0%	28.30	13.37
	WTC	0.1%	1.00	1.60
		1.0%	18.87	39.87
<u>Toluene</u>	WES	0.1%	3.03	4.43
		1.0%	55.43	93.67
	PCE	0.1%	1.37	2.50
		1.0%	36.67	35.77
	WTC	0.1%	1.24	1.39
		1.0%	65.67	89.57
<u>Ethylbenzene</u>	WES	0.1%	5.27	17.33
		1.0%	33.83	47.33
	PCE	0.1%	2.03	2.33
		1.0%	34.53	20.93
	WTC	0.1%	2.93	3.94
		1.0%	36.10	95.60

(Continued)

(Sheet 2 of 3)

TABLE 23. (Concluded)

Organic Contaminant	Sludge	Organic Level	Extract Concentration (mg/l)	
			EP	TCLP
<u>Butanone</u>	WES	0.1%	35.80	17.00
		1.0%	188.00	256.67
	PCE	0.1%	5.19	5.39
		1.0%	133.33	134.33
	WTC	0.1%	9.59	6.29
		1.0%	163.00	165.67
<u>4-Methyl-2-Pentanone</u>	WES	0.1%	41.33	13.33
		1.0%	192.67	313.33
	PCE	0.1%	11.63	10.63
		1.0%	233.00	247.00
	WTC	0.1%	7.67	4.88
		1.0%	298.00	306.00

(Sheet 3 of 3)

TABLE 24. RESULTS OF STATISTICAL ANALYSIS FOR NORMALIZED  
TCLP AND EP ORGANIC EXTRACT CONCENTRATIONS

Organic Constituent	Sludge*	Extraction Test†	Organic Level‡	Replicate	Extraction Test Inter- action	R Values§
Chloroform	Y	Y	Y	N	N	0.93
1,2-Dichloroethane	Y	N	Y	N	N	0.93
1,1,1-Trichloroethane	Y	Y	Y	N	Y	0.96
Carbon Tetrachloride	Y	N	Y	N	N	0.93
Trichloroethene	Y	Y	Y	N	Y	0.98
Benzene	Y	Y	Y	N	Y	0.98
1,1,2,2-Tetrachloroethane	Y	N	Y	N	N	0.98
Tetrachloroethene	Y	Y	Y	N	Y	0.99
Toluene	Y	Y	Y	N	Y	0.99
Ethylbenzene	Y	Y	Y	N	Y	0.95
2-Butanone	Y	N	Y	N	N	0.96
4-Methyl-2-Pentanone	Y	Y	Y	N	Y	0.98

Note: Results presented as "Yes" (Y) or "No" (N). Yes indicates that there is statistical difference between the variable compared at  $\alpha = 0.05$ .

\* WES, PCE, and WTC sludges.

† EP and TCLP.

‡ 0.1% and 1.0%.

§ R values give an indication of how well the statistical model fits the data. as the fit of the model improves, the R value approaches 1.0.

TABLE 25. RESULTS OF PAIRED-SAMPLE T TEST FOR  
NORMALIZED STUDY B TCLP AND EP ORGANIC  
EXTRACT CONCENTRATIONS

Organic Constituent	Extraction Test*
Chloroform	--
1,2-Dichloroethane	--
1,1,1-Trichloroethane	Y
Carbon Tetrachloride	--
Trichloroethene	Y
Benzene	Y
1,1,2,2-Tetrachloroethane	--
Tetrachloroethene	N
Toluene	Y
Ethylbenzene	Y
2-Butanone	--
4-Methyl-2-Pentanone	N

Note: The paired T test was performed only when the levels of interaction were found to be significant in the AVMFT.

\* Yes (Y) indicates there is statistical difference between the EP and TCLP results at  $\alpha = 0.05$ .

that, generally, the TCLP test generated extracts that contained higher concentrations of organic contaminants than the EP extracts. Higher concentrations of organics in the TCLP extracts were expected because the TCLP extraction was performed under zero-headspace conditions. However, the difference was not as great as expected.

Results of the AVMFT performed on the Study B organic data are presented in Table 24. As in Study A, when the results of the AVMFT indicated that the levels of test interaction are significant, a paired T test was performed. The results of the paired T test are presented in Table 25. The TCLP and EP extracts are statistically different for over half of the organic constituents evaluated. Statistical analysis for only six of the organic constituents (1,2-dichloroethane, carbon tetrachloride, 1,1,2,2-tetrachloroethane, tetrachloroethene, 2-butanone, and 4-methyl-2-pentanone) indicated no statistical difference between leach test extracts. Contaminant levels of two (1,1,2,2-tetrachloroethene and carbon tetrachloride) of the six organic constituents were near the detection limit. Consequently, 1,2-dichloroethane, tetrachloroethene, 2-butanone, and 4-methyl-2-pentanone were the only organics extracted from the waste equally by the EP and TCLP.

A graphical representation of the results of the TCLP and EP extractions for the organic compounds in Study B is presented in Appendix J, Figures J1 through J12. In these figures, the normalized EP extract concentrations are plotted versus the normalized TCLP extract concentrations. A discussion of the results accompanies these figures.

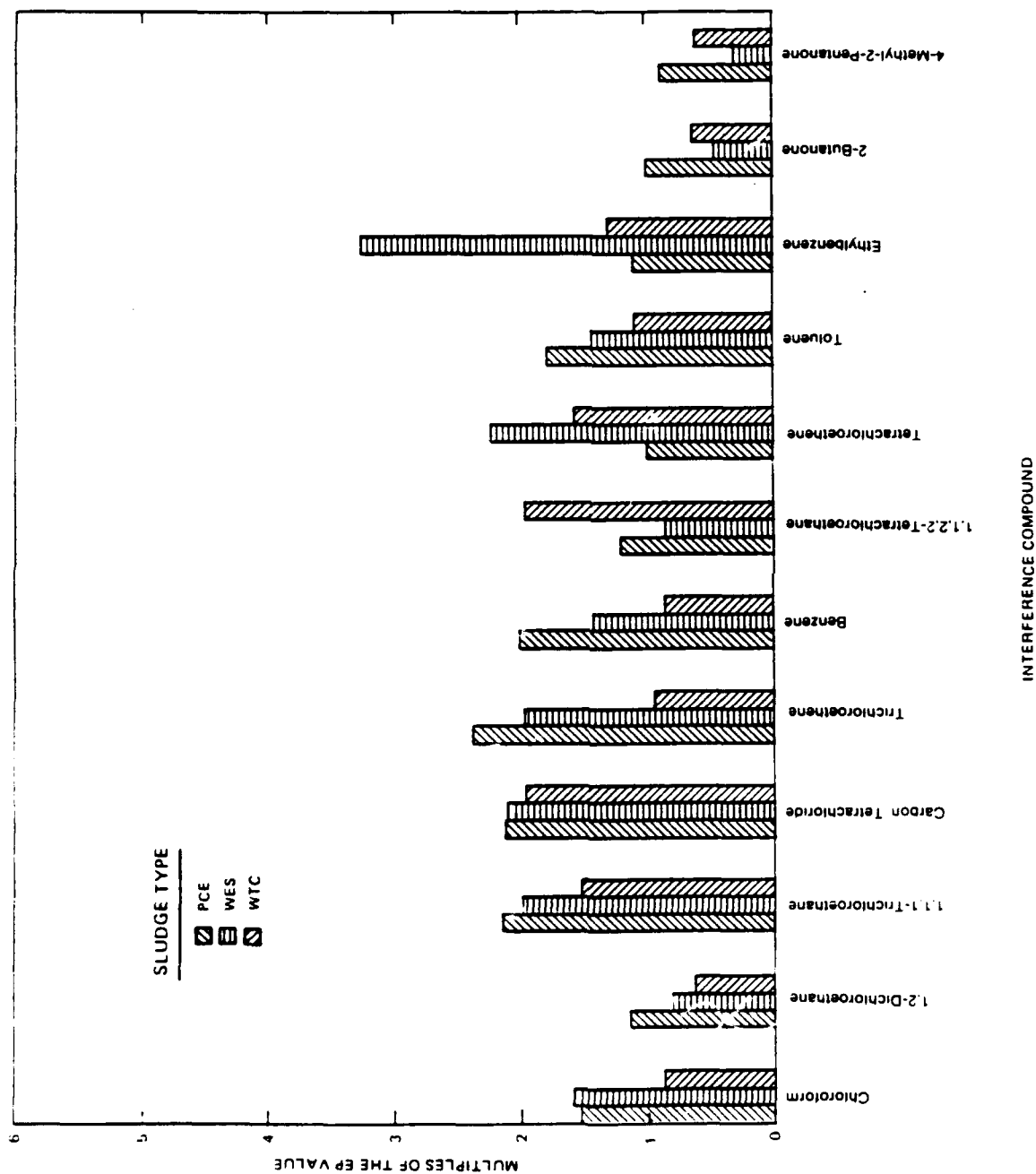


Figure 12. Average normalized Study B 0.1% organic extract concentrations expressed as the TClP concentration divided by the EP concentration.

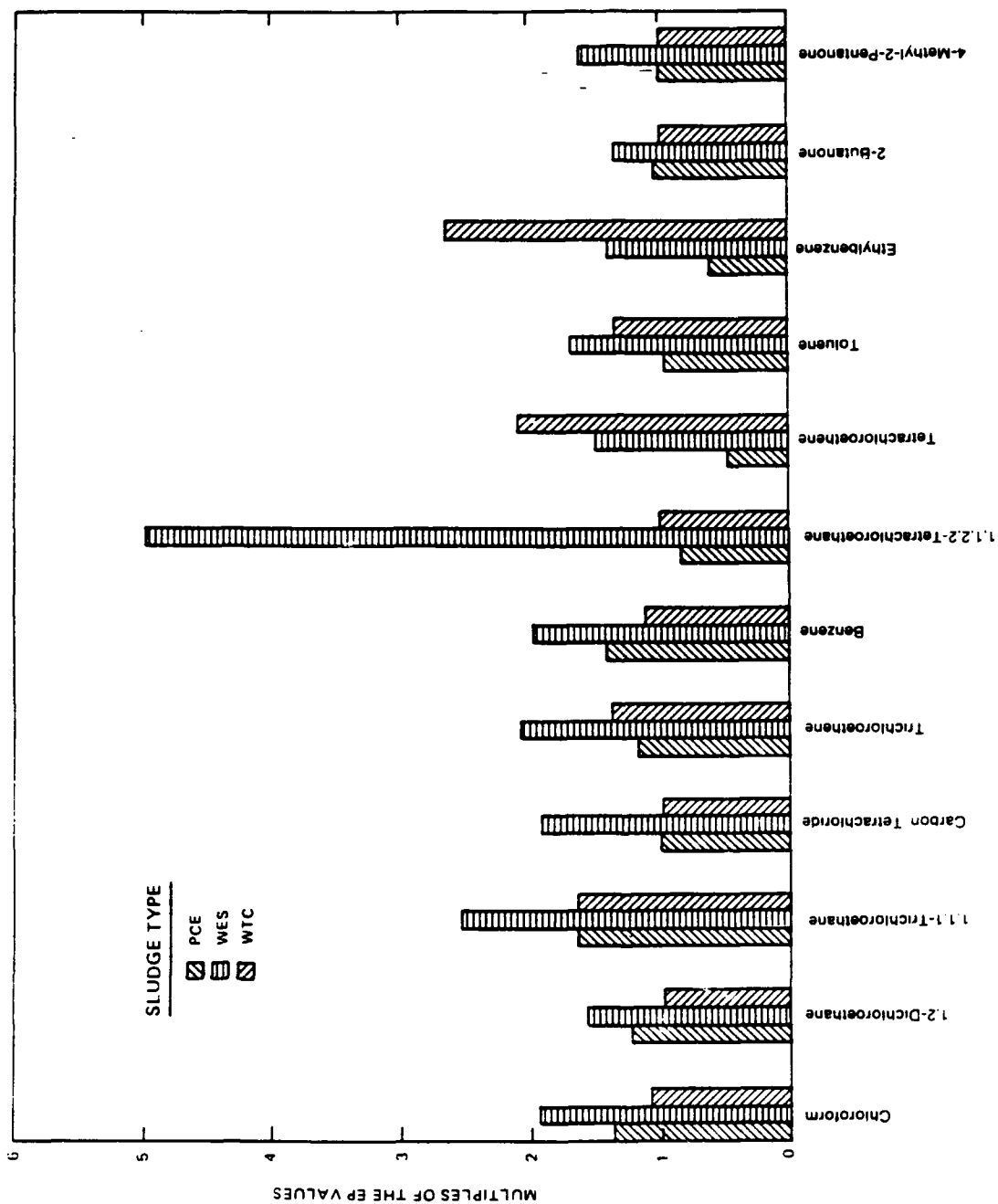


Figure 13. Average normalized Study B 1.0% organic extract concentrations expressed as the TCLP concentration divided by the EP concentration.

Figures 12 and 13 present the results for the 0.1% and 1.0% organic extracts (WES, PEC, and WTC). These figures show the normalized TCLP and EP extract expressed as multiples of the EP values, averaged for the three replicate specimens. These figures illustrate that, typically, TCLP organic extract concentrations are 1.5 times larger than those measured in the EP extracts. However, these figures also indicate some exceptions to this general finding. Compounds detected in the EP extracts at concentrations greater than 1.1 times the TCLP extracts included: 1,2-dichloroethane, benzene, 1,1,2,2-tetrachloroethane, 2-butanone, and 4-methyl-2-pentanone (for the 0.1% organic extracts) and tetrachloroethene and ethylbenzene for the 1.0% organic extracts.

Before this study was initiated, it was expected that the TCLP would generate extracts with much higher concentrations of organics than the EP extracts. As shown in Figures 12 and 13, the extracts from the TCLP have only slightly higher concentrations of organics than the organics measured in the EP extracts.

Another interesting observation is seen in the data presented in Table 26. This table presents the bulk analysis of the sludges immediately before the TCLP or EP extractions. The initial concentrations (the sludge concentration prior to extraction) of organics in the 1.0% sludges were 3.8 to 510 times greater than the initial organic concentrations of the 0.1% sludges. While up to 510 times more organics were originally present in the 1.0% sludge, the EP and TCLP produce extracts with organic concentrations only 1.5 times higher than the extract produced by the 0.1% sludge. It should be noted that if all the organic compounds were extracted from the sludges, the resulting organic/water mixture would be well below any solubility limits. One would expect the larger driving force in the 1.0% sludge to produce a more concentrated extract than the 0.1% sludge. However, this was not the case.

Attempts were made to correlate the data presented in Figures 11 and 12 with various physical properties such as vapor pressure, solubility, pH, and boiling point; however, no evidence of correlation with any of these variables was found. This refutes postulations such as (1) the more volatile compounds should be detected in the TCLP extracts at greater concentration than the EP extracts or (2) the difference in pH of the EP and TCLP extraction fluids could result in more extraction of the organic compounds from the waste. Due to the complex nature of the wastes and the many variables involved with the EP and TCLP extractions, no explanations are made to clarify why (in some cases) the EP generated leachates with higher concentrations of organics than the TCLP. It appears that vapor pressure, solubility, pH, and boiling point are not linked to this phenomenon.

#### SPIKE AND RECOVERY STUDY

The results of the spike and recovery study for samples that were prespiked are presented in Table 27, and the results for the postspike samples are presented in Table 28. The results in Tables 27 and 28 are presented as

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<sup>1</sup> Physical data for the organic compounds are presented in Table D-1, Appendix D.

TABLE 26. STUDY B ORGANIC SLUDGE BULK ANALYSES  
(PRESENTED ON WET AND DRY BASIS)

	0.1%		1.0%	
	Wet (mg/kg)	Dry (mg/kg)	Wet (mg/kg)	Dry (mg/kg)
<u>WES Sludge</u>				
Chloroform	70.3	35.9	6,443.6	3,212.8
1,2-Dichloroethane	264.4	134.9	11,248.8	5,608.6
1,1,1-Trichloroethane	62.0	31.6	18,581.4	9,264.7
Carbon Tetrachloride	62.6	31.9	7,572.4	3,775.6
Trichloroethene	221.7	113.1	42,257.7	21,069.7
Benzene	131.2	66.9	22,077.9	11,008.1
1,1,2,2-Tetrachloroethane	24.4	12.4	249.8	124.5
Tetrachloroethene	255.5	130.3	41,158.8	20,521.8
Toluene	232.6	118.7	33,066.9	16,487.2
Ethylbenzene	427.5	218.0	47,952.0	23,908.9
2-Butanone	3,707.9	1,891.4	47,052.9	23,460.6
4-Methyl-2-Pentanone	2,892.8	1,475.6	70,030.0	34,916.9
<u>PCE Sludge</u>				
Chloroform	217.7	169.6	6,337.3	4,512.8
1,2-Dichloroethane	445.4	347.0	9,769.6	6,956.9
1,1,1-Trichloroethane	328.6	256.0	8,516.0	6,064.3
Carbon Tetrachloride	194.8	151.7	4,536.6	3,230.5
Trichloroethene	870.9	678.5	17,808.1	12,681.1
Benzene	830.9	647.4	18,106.5	12,893.6
1,1,2,2-Tetrachloroethane	4,104.8	3,198.1	22,185.5	15,798.3
Tetrachloroethene	3,625.4	2,824.6	16,216.3	11,547.6
Toluene	729.1	568.0	18,902.4	13,460.4
Ethylbenzene	5,832.6	4,544.2	24,175.2	17,215.1
2-Butanone	485.4	378.2	25,667.5	18,277.8
4-Methyl-2-Pentanone	1,018.7	793.7	25,866.4	18,419.5
<u>WTC Sludge</u>				
Chloroform	2.9	2.4	576.4	458.4
1,2-Dichloroethane	13.1	10.8	1,931.4	1,535.8
1,1,1-Trichloroethane	4.2	3.4	2,180.3	1,733.7
Carbon Tetrachloride	1.2	1.0	333.5	265.2
Trichloroethene	44.6	36.7	9,537.4	7,584.1
Benzene	11.2	9.2	3,793.0	3,016.2
1,1,2,2-Tetrachloroethane	1.2	1.0	125.4	99.7
Tetrachloroethene	51.2	42.0	7,566.2	6,016.6
Toluene	28.9	23.7	6,590.5	5,240.8
Ethylbenzene	114.9	94.3	10,552.8	8,391.6
2-Butanone	192.5	158.0	3,195.7	2,541.2
4-Methyl-2-Pentanone	150.2	123.3	10,652.4	8,470.8

TABLE 27. AVERAGE PERCENT OF VOLATILES LOST FROM PRESPIKE SAMPLES

Sludge	Leach Test	Organic Level (percent)	Spike Compound	
			Chlorobenzene (percent)	Carbon Disulfide (percent)
WTC	TCLP	0.1	*	ND†
WTC	EP	0.1	*	99.24
WTC	TCLP	1	*	ND
WTC	EP	1	*	ND
PCE	TCLP	1	ND	ND
PCE	EP	1	ND	ND

\* Sample not spiked with analyte.

† Compound was below the detection limit; thus, not detected in the extract.

TABLE 28. AVERAGE PERCENT OF VOLATILES LOST FROM POSTSPIKE SAMPLES

Sludge	Leach Test	Organic Level (percent)	Spike Compound	
			Chlorobenzene (percent)	Carbon Disulfide (percent)
WTC	TCLP	0.1	23.53	*
WTC	EP	0.1	5.50	*
WTC	TCLP	1	4.58	*
WTC	EP	1	25.77	*
PCE	TCLP	0.1	8.62	23.60
PCE	EP	0.1	16.59	9.11

\* Sample not spiked with analyte.

the percent of spike compound lost from the extract. A problem encountered with the organic spikes was that the compounds used to spike the WES sludge extracts did not adequately disperse. While the problem was corrected for the chlorobenzene and carbon disulfide spikes, it was not corrected for the 1,1,2-trichloroethane spike. Consequently, the spike data for the WES sludge extracts and the 1,1,2-trichloroethane spike are omitted from this discussion.

#### Prespike Extracts

Results of the prespike extracts (Table 27) indicate that greater than 99 percent of the compounds used as spikes were lost both from the TCLP and EP extracts. These losses of the prespike chlorobenzene and carbon disulfide can be explained either by (1) absorption of these compounds by the solid waste used in the extraction or (2) loss of these compounds from the EP and TCLP extracts during the extraction process.

#### Postspike Extracts

##### Chlorobenzene--

Results of the triplicate extracts postspiked with chlorobenzene were statistically evaluated using an A by B two-way classification analysis of the variance technique (Miller and Freund 1985). Results of this analysis indicate that, at an alpha level of significance of 0.05, there is no statistical evidence that either the replication, tests (EP or TCLP), or sludges (WTC-0.1%, WTC-1.0%, or PCE-0.1%) differ. These results were expected based on the fact that there was no variation in any of the extraction methods after the postspike was injected into the extract sample.

##### Carbon Disulfide--

Results of the triplicate extracts postspiked with carbon disulfide were also statistically evaluated. In this case only two conditions were compared, the EP and TCLP for the PCE sludge at 0.1% organic level. These samples were compared using a student "T" test (Miller and Freund 1985). Results from this analysis indicated that, at an alpha level of significance of 0.05, there was no statistical evidence that the amount of spike lost from the EP extracts differed from the spike lost from the TCLP extracts.

#### Summary

Although there was little difference between loss of postspike compounds from the extracts, the postspike data yield some useful information. First, in the worst case, a maximum of 25% of the volatile spike was lost during sample placement into the sample vial, storage, and analysis. Second, the high recoveries observed for the postspiked sample for chlorobenzene and carbon disulfide indicate that these materials probably were well dispersed. Thus, the large prespike losses cannot be attributed to poor sample dispersion.

## QUALITY ASSURANCE/QUALITY CONTROL

The results of the method blanks for the Study A metal analyses are presented in Table 29; for the Study B metal analyses in Table 30; and for the Study B volatile organic analyses in Table 31. The method blanks for both Study A and B metal analyses indicate that some of the contaminants are detected in the method blanks; however, for the majority of the samples that were analyzed, the method blanks are relatively uncontaminated (excluding nickel). Although nickel concentrations 10 times the detection limit are detected in the method blanks, no method blank corrections for nickel, or any metal compounds, are made. This decision is based on the fact that the concentrations of most of the metal compounds are well above the detection limits.

The results of the method blanks for the Study B volatile organics data indicate that, for many contaminants, the concentration of organics detected in the blank extracts is well above the detection limit. This indicates that some residual contamination of the extraction media is occurring. It is suspected that this contamination may be the result of residual left in the ZHE apparatus, although many precautions were taken to prevent such contamination.

Results of the internal QA/QC are presented in Tables 32 through 35. As indicated in these tables, the internal QA/QC was excellent.

Results of the external QA/QC are presented in Tables 36 and 37. The results of the external sample do not reflect the level of quality indicated by the internal QA/QC. However, except for some of the mercury data, the external QA/QC data represent a relatively high degree of quality throughout this study.

## PROCEDURAL DIFFICULTIES ENCOUNTERED WITH THE TCLP

The TCLP extraction is more difficult to perform than the EP extraction. Factors that contribute to the difficulty include:

- (1) The TCLP requires two extractions, one for volatiles and another for nonvolatiles. The EP only requires one extraction.
- (2) The TCLP uses two extraction fluids and requires a prescreening test to determine which extraction fluid to use. The EP requires one extraction fluid.
- (3) The TCLP ZHE vessel is difficult to clean, as illustrated by the high degree of contamination in the ZHE blanks (Table 31). It is suspected that the valve on the ZHE may trap small amounts of liquid which may contaminate subsequent extractions.
- (4) The TCLP method does not provide clear directions on the use of volatile organic vials for extract collection. Since the sample must be exposed to the atmosphere during sample collection, incorrect sample handling may result in large volatile organic losses.

TABLE 29. ANALYSIS OF METHOD BLANKS FOR THE METALS STUDY A TCLP/EP TEST

Interference Compound	Test	Study A Metal Contaminants (mg/l)			
		Cd	Cr	Ni	Hg
Copper	EP	0.0007	0.0170	0.0230	<0.0004
	TCLP	<0.0001	0.0010	0.0020	<0.0004
Grease	EP	0.0048	0.0080	0.0070	<0.0008
	TCLP	<0.0001	0.0080	0.0070	<0.0008
Hexachlorobenzene	EP	0.0005	0.0140	0.0090	<0.0004
	TCLP	<0.0001	0.0080	0.0080	<0.0004
Sodium sulfate	EP	0.0008	0.0030	0.0140	<0.0004
	TCLP	0.0002	0.0010	0.0200	<0.0004
Sodium hydroxide	EP	0.0004	0.0060	0.0080	<0.0008
	TCLP	<0.0001	0.0030	0.0310	<0.0008
Oil	EP	<0.0001	0.0230	0.0040	<0.0008
	TCLP	0.0206	0.0110	0.0140	<0.0008
Lead	EP	0.0009	0.0270	0.0280	<0.0008
	TCLP	0.0001	0.0270	0.0280	<0.0008
Phenol	EP	0.0002	0.0080	0.0020	<0.0004
	TCLP	<0.0001	0.0050	0.0050	<0.0004
Trichloroethene	EP	0.0087	0.0690	0.0780	<0.0004
	TCLP	<0.0001	0.0020	0.0140	<0.0004
Zinc	EP	0.0014	0.0060	0.0260	<0.0008
	TCLP	0.0009	0.0020	0.0010	<0.0008

(5) When the extraction fluid is added to the ZHE apparatus, it is difficult to accurately measure the volume of extraction fluid. Pumping from a graduated cylinder offers one solution, but the large open area of the cylinder may permit contamination of the extraction fluid.

(6) An interesting phenomenon uncovered from the organic results of the TCLP and EP was the high concentrations of 1,1-dichloroethene (1,1-DCE) measured in the extracts. Although the sludges were not fortified with 1,1-DCE and no measurable concentrations of 1,1-DCE were detected in the bulk analyses of the raw sludges, high concentrations of 1,1-DCE were detected in both the TCLP and EP extracts. As shown in Table 38, only the extracts for the sludges that were solidified/stabilized had measurable concentrations of 1,1-DCE. It is suspected that some form of dechlorination reaction that favors the extract conditions of the solidified/stabilized materials is producing the 1,1-DCE. Similar phenomena have been reported by other researchers (Newcomer, Blackburn, and Kimmell 1986). While it is beyond the scope of this study to pinpoint the mechanism of the conversion reaction, this is a significant

TABLE 30. ANALYSIS OF METHOD BLANKS FOR THE METALS STUDY B TCLP/EP TEST

Sludge	Test	Organic Concen- tration	Study B Metals Contaminants (mg/l)										
			Antimony	Arsenic	Barium	Cadmium	Chromium	Copper	Lead	Mercury	Nickel	Silver	Zinc
WES	TCLP	0.12	NA*	NA	NA	0.0011	0.0100	NA	NA	<0.0008	0.0120	NA	NA
		1.02	NA	NA	NA	0.0018	0.0110	NA	NA	<0.0008	0.0360	NA	NA
	EP	0.12	NA	NA	NA	0.0051	0.0190	NA	NA	<0.0008	0.0200	NA	NA
		1.02	NA	NA	NA	0.0113	0.0490	NA	NA	0.0014	0.0110	NA	NA
WTC	TCLP	0.12	NA	<0.0050	NA	<0.0001	0.0050	NA	0.0120	NA	NA	NA	NA
		1.02	NA	<0.0050	NA	0.0006	0.0040	NA	0.0080	NA	NA	NA	NA
	EP	0.12	NA	<0.0050	NA	0.0003	0.0030	NA	0.0070	NA	NA	NA	NA
		1.02	NA	<0.0050	NA	<0.0001	0.0040	NA	0.0080	NA	NA	NA	NA
PTE	TCLP	0.12	<0.0050	<0.0050	0.0520	NA	NA	0.0030	<0.0010	NA	NA	0.0270	0.0640
		1.02	<0.0050	<0.0050	0.0530	NA	NA	0.0070	0.0020	NA	NA	0.0090	0.0740
	EP	0.12	<0.0050	<0.0050	0.0820	NA	NA	0.0060	0.0460	NA	NA	0.0010	0.0410
		1.02	<0.0050	<0.0050	0.1630	NA	NA	0.0100	0.0010	NA	NA	<0.0010	0.0710
Detection Limit			<0.0050	<0.0050	<0.001	<0.0001	<0.001	<0.0001	<0.001	<0.0008	<0.001	<0.001	<0.003

<sup>a</sup> Not analyzed.

TABLE 31. ANALYSIS OF METHOD BLANKS FOR THE VOLATILE ORGANICS STUDY B TCLP/EP TEST

Sludge	Test	Organic Fortification Concentration	Organic Contaminant (mg/l)												
			1,1-DCE	CHCL3	1,2-DCA	1,1,1-TCA	CCL4	TCE	BENZ	TCLTA	TCLTE	TOLUE	ETBEN	2-BUTA	4-MELPE
WES	EP	0.12	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.005	0.005	0.005	0.010	0.010
		1.02	<0.005	<0.005	<0.005	<0.005	0.028	<0.005	<0.005	0.026	0.016	0.014	0.010	0.010	
WTC	TCLP	0.12	<0.005	<0.005	<0.005	<0.005	<0.005	0.010	<0.005	<0.005	0.005	0.005	0.010	0.010	0.010
		1.02	0.140	0.015	0.028	0.067	0.033	0.440	0.150	0.005	0.370	0.390	0.400	0.050	0.190
PCE	EP	0.12	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.005	0.005	0.010	0.010	0.010
		1.02	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.005	0.005	0.005	0.005	0.029	0.010
	TCLP	0.12	<0.005	<0.005	<0.005	0.008	<0.005	0.018	0.014	<0.005	0.018	0.028	0.012	0.031	0.034
		1.02	<0.005	0.018	0.065	0.210	0.015	0.990	0.310	<0.005	1.050	0.980	2.000	0.076	0.360
	EP	0.12	<0.005	<0.005	<0.005	<0.005	<0.005	0.003	<0.005	<0.005	0.002	0.005	0	0.010	0.010
		1.02	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.005	0.005	0.005	0.010	0.010
	TCLP	0.12	0.076	0.005	0.011	0.037	0.015	0.140	0.056	<0.005	0.099	0.140	0.340	0.010	0.170
		1.02	0.005	0.071	0.098	0.038	0.028	0.210	0.280	0.077	0.130	0.240	0.200	0.061	0.190
Detection Limit			<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.005	0.005	0.005	0.01	0.01

1,1-DCE = 1,1-Dichloroethene	TCE = Trichloroethene	TCLTE = Tetrachloroethene	2-BUTA = 2-Butanone
CHCL3 = Chloroform	BENZ = Benzene	TOLUE = Toluene	4-MELPE = 4-Methyl-2-Pentanone
1,2-DCA = 1,2-Dichloroethane	TCLTA = 1,1,2,2-Tetrachloroethane	ETBEN = Ethylbenzene	
1,1,1-TCA = 1,1,1-Trichloroethane			
CCL4 = Carbon Tetrachloride			

TABLE 32. STUDY A METALS PERCENT ACCURACY OF THE ANALYTICAL LABORATORY'S  
INTERNAL STANDARDS

Interference Compound	NBS* Traceable Internal Standard			
	Cadium	Chromium	Nickel	Mercury
	(Percent Accuracy)			
Oil	98.5	94.3	98.3	94.6
Grease	89.6	97.1	96.6	98.0
Lead	98.5	94.3	99.2	94.6
Copper	94.0	91.4	90.4	98.7
Zinc	95.5	95.7	93.3	97.4
Sodium hydroxide	97.0	98.6	97.1	97.4
Sodium sulfate	73.1	89.3	94.6	98.7
Phenol	83.6	95.7	92.5	98.0
Hexachlorobenzene	85.1	94.3	95.4	100.0
Trichloroethene	91.0	98.6	93.3	94.6

\* National Bureau of Standards.

TABLE 33. STUDY B METALS PERCENT ACCURACY OF ANALYTICAL LABORATORY'S  
INTERNAL STANDARDS

Type of Waste	Contaminant	Standards	Standard Accuracy
WTC	Arsenic	A	98.0
		B	96.6
	Cadmium	A	93.4
	Lead	B	97.1
	Chromium	A	80.0
		B	95.6
		C	97.0
		D	97.0
WES	Cadmium	A	92.2
		B	92.2
	Chromium	A	89.4
	Nickel	A	98.6
		B	97.5
		C	97.5
	Mercury	A	96.8
PCE	Arsenic	A	94.3
		B	100.0
	Antimony	A	81.1
	Copper	A	95.0
		B	99.0
		C	99.0
	Lead	A	86.2
		B	86.2
	Silver	A	85.7
		B	85.7
	Barium	A	90.7
		B	92.2
		C	90.7
	Zinc	A	97.7
		B	97.7

TABLE 34. STUDY B ORGANIC INTERNAL SURROGATE SPIKES

Sludge	Test	Organic Level	Replicate	Surrogate Spike		BFB†	
				Toluene D8	1-2-DCA D4*		
(Percent Recovery)							
WES	TCLP	0.1%	R1	94.9	90.8	96.0	
			R2	108.0	103.0	114.0	
			R3	98.5	82.4	112.0	
			BL‡	97.3	80.7	105.0	
		1.0%	R1	97.6	118.0	104.0	
			R2	100.0	83.0	90.0	
			R3	100.0	108.0	101.0	
			EP	0.1%	R1	99.2	102.0
	R2	100.0			98.5	112.0	
	R3	107.0			96.0	96.6	
	BL	105.0			98.1	113.0	
	1.0%	R1		108.0	99.9	89.0	
		R2		109.0	94.4	93.6	
		R3		100.0	102.0	108.0	
		WTC		TCLP	0.1%	R1	97.0
	R2		100.0			119.0	100.0
R3	97.0		99.0			102.0	
BL	96.4		93.1			90.9	
1.0%	R1		101.0		100.0	98.7	
	R2		105.0		93.4	102.0	
	R3		97.1		100.0	103.0	
	EP		0.1%		R1	102.0	90.1
R2				102.0	90.8	99.3	
R3				99.2	94.4	100.0	
BL				109.0	93.0	100.0	
1.0%	R1		96.2	93.0	98.8		
	R2	99.7	92.7	95.8			
	R3	98.6	98.4	100.0			
	PCE	EP	0.1%	R1	101.0	89.3	103.0
R2				95.8	97.6	94.4	
R3				98.4	102.0	96.3	
PCE	EP	1.0%	R1	101.0	95.0	100.0	
			R2	99.7	92.8	98.5	
			R3	99.2	93.8	99.9	
	TCLP	0.1%	R1	99.4	100.0	97.3	
			R2	101.0	102.0	87.1	
			R3	99.3	91.7	101.0	
	TCLP	1.0%	R1	99.7	98.8	96.5	
			R2	97.2	99.2	92.6	
			R3	96.7	96.2	<93.3	

\* 1-2-Dichloroethane D4.

† Bromofluorobenzene.

‡ Blank.

TABLE 35. STUDY B ORGANIC DUPLICATE AND PERCENT RECOVERY ANALYSES

Waste	Organic Level	Extraction Test	Replicate	Contaminant					
				DCLETE*		CHCL3†		1,2-DCE‡	
				Duplicate	% Recovery	Duplicate	% Recovery	Duplicate	% Recovery
PCE	1.0%	TCLP	R1	25.0	NA§	37.1	NA	79.3	NA
	1.0%	TCLP	R2	10.0	NA	33.8	NA	69.7	NA
	1.0%	TCLP	R3	10.0	104.0	33.0	105.0	77.8	111.7
	1.0%	EP	R1	10.0	NA	20.4	NA	50.1	NA
	1.0%	EP	R3	10.0	101.9	28.0	101.8	74.8	77.7
WES	0.1%	TCLP	R3	175.0	NA	NA	NA	NA	NA
	0.1%	TCLP	R1	NA	NA	NA	NA	NA	NA
	1.0%	TCLP	R1	191.0	NA	18.0	NA	63.0	NA
	0.1%	EP	R2	NA	NA	NA	98.9	NA	99.8
	1.0%	EP	R3	77.7	NA	14.2	NA	40.1	NA
WTC	0.1%	EP	R3	5.0	NA	8.9	102.4	44.8	100.6
	1.0%	TCLP	R3	NA	102.0	NA	93.0	NA	92.7

(Continued)

\* 1,1-Dichloroethene.

† Chloroform.

‡ 1,2-Dichloroethane.

§ Not analyzed.

(Sheet 1 of 6)

TABLE 35 (Continued)

Waste	Organic Level	Extraction Test	Replicate	CCL4*		Contaminant TCE†		1,1,2-Trichloroethane‡	
				Duplicate	% Recovery	Duplicate	% Recovery	Duplicate	% Recovery
PCE	1.0Z	TCLP	R1	25.0	NA	38.8	NA	25.0	NA
	1.0Z	TCLP	R2	10.0	NA	37.5	NA	10.0	NA
	1.0Z	TCLP	R3	10.0	NA	43.5	105.3	10.0	109.5
	1.0Z	EP	R1	10.0	NA	33.4	NA	13.0	NA
	1.0Z	EP	R3	10.0	102.4	43.3	113.5	10.0	100.1
WES	0.1Z	TCLP	R3	NA	NA	5.9	NA	NA	NA
	0.1Z	TCLP	R1	NA	NA	NA	NA	NA	NA
	1.0Z	TCLP	R1	5.0	NA	140.0	NA	5.0	NA
	0.1Z	EP	R2	NA	102.0	4.1	100.0	NA	96.4
	1.0Z	EP	R3	4.3	NA	63.2	NA	1.0	NA
WTC	0.1Z	EP	R3	5.0	100.0	89.7	98.7	5.0	NA
	1.0Z	TCLP	R3	NA	84.7	NA	100.6	NA	107.0

(Continued)

\* Carbon tetrachloride.

† Trichloroethene.

‡ 1,1,2-Trichloroethane.

(Sheet 2 of 6)

TABLE 35 (Continued)

Waste	Organic Level	Extraction Test	Replicate	Contaminant					
				TCLETA*		TCLETE†		TOLUENE‡	
				Duplicate	% Recovery	Duplicate	% Recovery	Duplicate	% Recovery
PCE	1.0%	TCIP	R1	80.6	NA	25.0	NA	35.7	NA
	1.0%	TCIP	R2	73.7	NA	13.9	NA	37.0	NA
	1.0%	TCIP	R3	91.1	84.7	17.0	87.7	39.1	86.0
	1.0%	EP	R1	93.0	NA	27.6	NA	35.4	NA
	1.0%	EP	R3	86.7	80.5	27.2	89.4	39.6	91.7
WES	0.1%	TCIP	R3	NA	NA	5.6	NA	3.6	NA
	0.1%	TCIP	R1	NA	NA	NA	NA	NA	NA
	1.0%	TCIP	R1	5.0	NA	37.0	NA	98.0	NA
	0.1%	EP	R2	NA	102.0	NA	104.5	3.5	93.8
	1.0%	EP	R3	1.0	NA	26.2	NA	62.8	NA
WTC	0.1%	EP	R3	5.0	96.3	19.7	NA	65.9	96.1
	1.0%	TCIP	R3	NA	101.0	NA	96.4	NA	96.1

(Continued)

\* 1,1,2,2-Tetrachloroethane.

† Tetrachloroethene.

‡ Toluene.

(Sheet 3 of 6)

TABLE 35 (Continued)

Waste	Organic Level	Extraction Test	Replicate	Contaminant					
				ETBEN*		2-BUTAT†		4ME2PE‡	
				Duplicate	% Recovery	Duplicate	% Recovery	Duplicate	% Recovery
PCE	1.0Z	TCLP	R1	25.0	NA	155.0	NA	256.0	NA
	1.0Z	TCLP	R2	20.8	NA	131.0	NA	219.0	NA
	1.0Z	TCLP	R3	22.3	101.2	132.0	NA	236.0	NA
	1.0Z	EP	R1	34.1	NA	117.0	NA	217.0	NA
	1.0Z	EP	R3	35.8	97.4	144.0	91.1	229.0	73.7
WES	0.1Z	TCLP	R3	NA	NA	NA	NA	NA	NA
	0.1Z	TCLP	R1	NA	NA	NA	NA	NA	NA
	1.0Z	TCLP	R1	50.0	NA	100.0	NA	94.0	NA
	0.1Z	EP	R2	5.1	101.8	108.0	97.8	106.0	91.4
	1.0Z	EP	R3	35.2	NA	108.0	NA	98.1	NA
WTC	0.1Z	EP	R3	35.8	93.8	201.0	NA	298.0	96.7
	1.0Z	TCLP	R3	NA	95.7	NA	102.0	NA	101.7

(Continued)

\* Ethylbenzene.

† 2-Butanone.

‡ 4-Methyl-2-Pentanone.

(Sheet 4 of 6)

TABLE 35 (Continued)

Waste	Organic Level	Extraction Test	Replicate	1,1,1-TCA*				Contaminant BENZENE†			
				Duplicate		% Recovery		Duplicate		% Recovery	
PCE	1.0%	TCLP	R1	25.7	NA	NA	77.8	NA	25.0	NA	NA
	1.0%	TCLP	R2	23.1	NA	NA	77.4	NA	10.0	NA	NA
	1.0%	TCLP	R3	29.5	108.3	102.5	79.8	102.5	10.0	103.3	103.3
	1.0%	EP	R1	12.6	NA	NA	49.3	NA	10.0	NA	NA
	1.0%	EP	R3	25.4	99.1	111.2	69.5	111.2	10.0	99.2	99.2
WES	0.1%	TCLP	R3	NA	NA	NA	NA	NA	8.0	NA	NA
	0.1%	TCLP	R1	NA	NA	NA	NA	NA	NA	NA	NA
	1.0%	TCLP	R1	44.0	NA	NA	84.0	NA	5.0	NA	NA
	0.1%	EP	R2	NA	NA	97.5	NA	97.5	NA	NA	NA
	1.0%	EP	R3	19.0	NA	NA	44.3	NA	11.1	NA	NA
WTC	0.1%	EP	R3	15.1	NA	93.4	57.2	93.4	5.0	98.2	98.2
	1.0%	TCLP	R3	NA	87.5	98.5	NA	98.5	NA	98.0	98.0

(Continued)

\* 1,1,1-Trichloroethane.  
† Chlorobenzene.

(Sheet 5 of 6)

TABLE 35 (Concluded)

Waste	Organic Level	Extraction Test	Replicate	Contaminant CS2*	
				Duplicate	% Recovery
PCE	1.0Z	TCLP	R1	<25.0	NA
	1.0Z	TCLP	R2	<10.0	NA
	1.0Z	TCLP	R3	<10.0	NA
	1.0Z	EP	R1	<10.0	NA
	1.0Z	EP	R3	<10.0	105.2
WES	0.1Z	TCLP	R3	NA	NA
	0.1Z	TCLP	R1	NA	NA
	1.0Z	TCLP	R1	101.0	NA
	0.1Z	EP	R2	113.0	98.2
	1.0Z	EP	R3	90.9	NA
WTC	0.1Z	EP	R3	<5.0	NA
	1.0Z	TCLP	R3	NA	109.2

\* Carbon disulfide.

(Sheet 6 of 6)

TABLE 36. STUDY A METALS PERCENT ACCURACY OF THE EXTERNAL STANDARDS

Extraction Test	Interference Compound	External Standard			
		Cadium	Chromium	Nickel	Mercury
TCLP	Oil	90.3	74.6	95.0	<0.16
EP		103.6	94.4	98.1	<0.32
TCLP	Grease	100.0	94.6	100.7	90.0
EP		112.0	96.4	101.7	53.0
TCLP	Lead	103.4	96.8	99.5	<0.16
EP		99.6	92.0	95.9	<0.32
TCLP	Copper	106.4	101.2	104.9	70.0
EP		108.8	102.4	98.4	64.5
TCLP	Zinc	102.0	97.6	101.1	67.0
EP		95.5	81.0	96.9	96.0
TCLP	Sodium hydroxide	104.0	88.4	100.4	60.0
EP		118.0	112.0	102.7	57.0
TCLP	Sodium sulfate	10.1	75.4	101.6	49.0
EP		104.6	94.0	102.1	188.0
TCLP	Phenol	87.4	87.2	101.7	67.6
EP		105.4	86.0	100.8	58.0
TCLP	Hexachlorobenzene	102.8	96.8	97.5	42.0
EP		90.5	83.0	99.7	56.0
TCLP	Trichloroethene	90.0	88.8	97.2	67.0
EP		110.0	106.4	103.9	140.0

factor that must be considered. If the contaminants of interest in the solidified/stabilized waste are converted to 1,1-DCE during the extraction, the concentration of 1,1-DCE in the extracts must be measured. If 1,1-DCE is an omitted parameter, large concentrations of volatile contaminants leaching from the solidified/stabilized waste will remain undetected. This could eventually result in long-term environmental degradation. Additional research is needed to clarify this issue.

TABLE 37. STUDY B METALS PERCENT ACCURACY OF  
EXTERNAL STANDARDS

Type of Waste	Contaminant	Percent Accuracy
WES	Cadmium	98.0
	Chromium	90.4
	Nickel	99.8
	Mercury	82.6
WTC	Arsenic	NA*
	Cadmium	NA
	Chromium	NA
	Lead	NA
PCE	Antimony	NA
	Arsenic	NA
	Barium	88.8
	Copper	NA
	Lead	68.4
	Silver	NA
	Zinc	NA

Not analyzed.

TABLE 38. CONCENTRATION OF 1,1-DICHLOROETHENE MEASURED  
IN THE TCLP AND EP EXTRACTS

Sludge	Extraction Test	Concentration	Extract Concentration (mg/l)
WES	EP	0.1%	67.77
		1.0%	92.10
	TCLP	0.1%	175.00
		1.0%	183.00
PCE	EP	0.1%	<0.33
		1.0%	<10.00
	TCLP	0.1%	<0.50
		1.0%	<10.00
WTC	EP	0.1%	4.05
		1.0%	<5.00
	TCLP	0.1%	9.94
		1.0%	<5.00

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## APPENDIX A

### EXTRACTION PROCEDURE (EP) TOXICITY TEST AND STRUCTURAL INTEGRITY TEST\*

#### 1.0 Scope and Application

1.1 The extraction procedure (EP) described in this method is designed to simulate the leaching a waste will undergo if disposed of in an improperly designed sanitary landfill. Method 1310 is applicable to liquid, solid, and multiphasic samples.

#### 2.0 Summary of Method

2.1 If a representative sample of the waste contains more than 0.5% solids, the solid phase of the sample is extracted with deionized water which is maintained at a pH of  $5 \pm 0.2$  using acetic acid. The extract is analyzed to determine if any of the threshold limits listed in Table A-1 are exceeded. Table A-1 also specifies the approved method of analysis. Wastes that contain less than 0.5% solids are not subjected to extraction, but are directly analyzed and evaluated in a manner identical to that of extracts.

#### 3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods referenced in Table A-1.

#### 4.0 Apparatus and Materials

4.1 Extractor: For purposes of this test, an acceptable extractor is one that will impart sufficient agitation to the mixture to (1) prevent stratification of the sample and extraction fluid and (2) ensure that all sample surfaces are continuously brought into contact with well-mixed extraction fluid. Examples of suitable extractors are shown in Figures A-1 through A-3 of this method and are available from Associated Design and Manufacturing Co., Alexandria, VA; Glas-Col Apparatus Co., Terre Haute, IN; Millipore, Bedford, MA; and Rexnord, Milwaukee, WI.

4.2 pH meter or pH controller: Chemtrix, Inc., Hillsboro, OR, is a possible source of a pH controller.

4.3 Filter holder: A filter holder capable of supporting a 0.45- $\mu$  filter membrane and able to withstand the pressure needed to accomplish separation. Suitable filter holders range from simple vacuum units to relatively complex systems that can exert up to 75 psi of pressure. The type of filter holder used depends upon the properties of the mixture to be filtered. Filter holders known to EPA and deemed suitable for use are listed in Table A-2.

4.4 Filter membrane: Filter membrane suitable for conducting the required filtration shall be fabricated from a material that (1) is not

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\* Source: U.S. Environmental Protection Agency, 1982. "Test Methods for Evaluating Solid Waste," SW-846, 2nd ed., Office of Solid Waste and Emergency Response, Washington, DC.

TABLE A-1. MAXIMUM CONCENTRATION OF CONTAMINANTS  
FOR CHARACTERISTIC OF EP TOXICITY

Contaminant	Maximum Concentration (mg/l)	Analytical Method
Arsenic	5.0	7060, 7061
Barium	100.0	7080, 7081
Cadmium	1.0	7130, 7131
Total chromium	5.0	7190, 7191
Hexavalent chromium	5.0	7195, 7196, 7197
Lead	5.0	7420, 7421
Mercury	0.2	7470
Selenium	1.0	7740, 7741
Silver	5.0	7760, 7761
Endrin (1,2,3,4,10,10-Hexachloro-1 7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1 4-endo, endo-5,8-dimethanonaphthalene)	0.02	8080
Lindane (1,2,3,4,5,6- Hexachlorocyclohexane, gamma isomer)	0.4	8080
Methoxychlor (1,1,1-Trichloro-2,2-bis (p-methoxyphenyl)ethane)	10.0	8080
Toxaphene (C <sub>10</sub> H <sub>10</sub> C <sub>18</sub> , Technical chlorinated camphene, 67-69% chlorine)	0.5	8080
2,4-D (2,4-Dichlorophenoxyacetic acid)	10.0	8150
2,4,5-TP (Silvex) (2,4,5- Trichlorophenoxypropionic acid)	1.0	8150

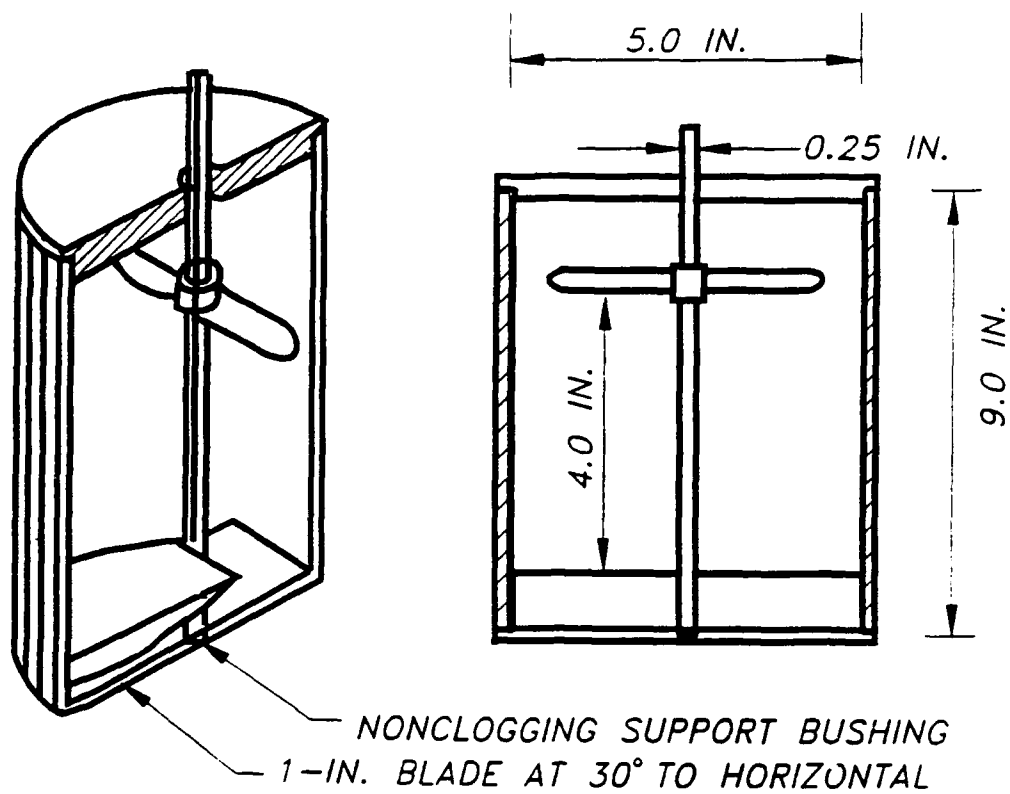


Figure A-1. EP extractor.

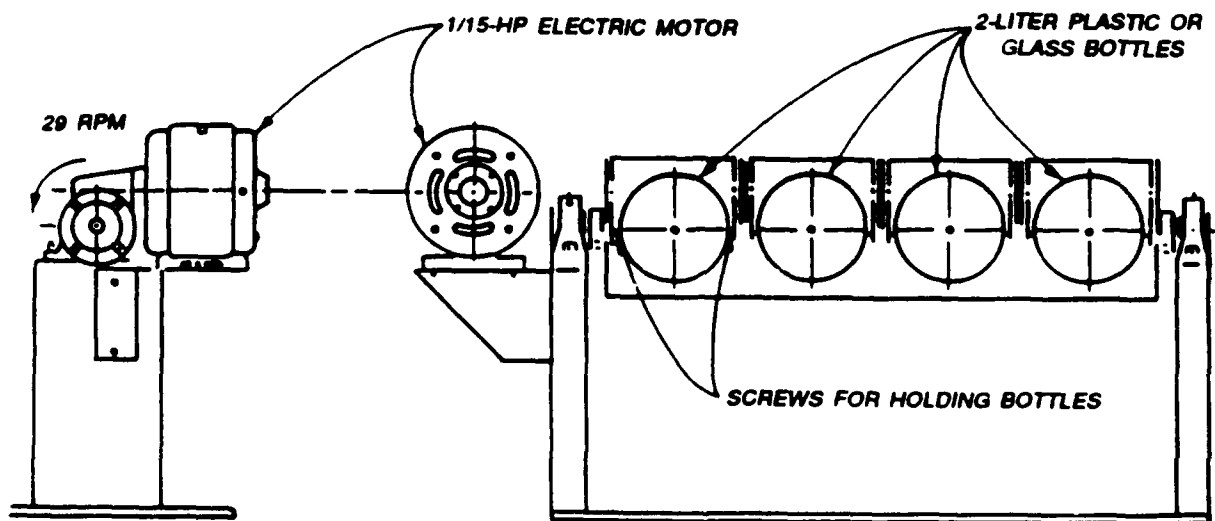


Figure A-2. EP rotary extractor.

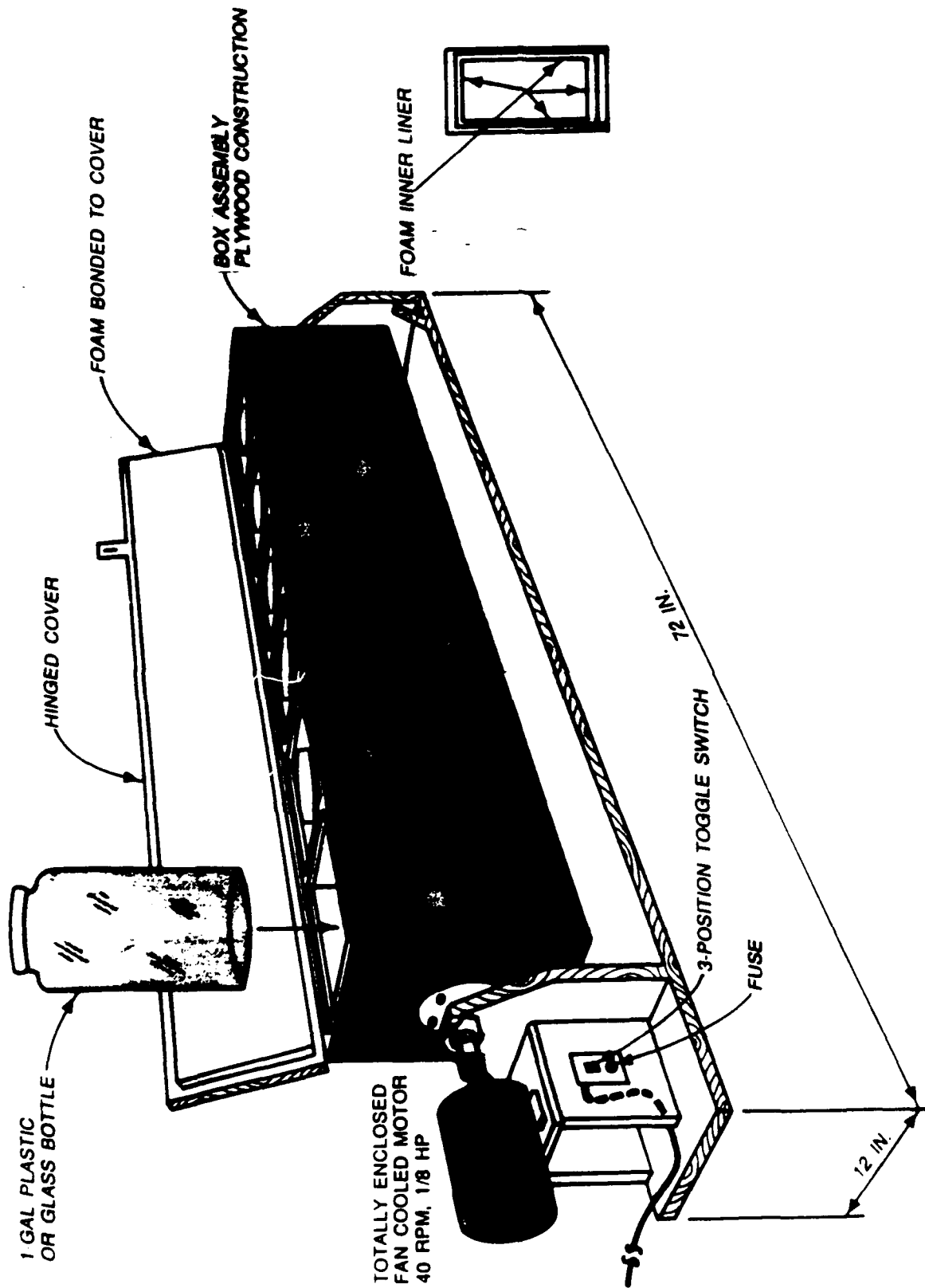


Figure A-3. EP EPRI extractor.

TABLE A-2. EPA-APPROVED FILTER HOLDERS

Manufacturer	Size	Model No.	Comments
<u>Vacuum filters</u>			
Nalgene	500 ml	44-0045	Disposable plastic unit, includes prefilter and filter pads, and reservoir; should be used when solution is to be analyzed for inorganic constituents
Nuclepore	47 mm	410400	
Millipore	47 mm	XX10 047 00	
<u>Pressure filters</u>			
Nuclepore	142 mm	425900	
Micro Filtration Systems	142 mm	302300	
Millipore	142 mm	YT30 142 HW	

physically changed by the waste material to be filtered and (2) does not absorb or leach the chemical species for which a waste's EP Extract will be analyzed. Table A-3 lists filter media known to the agency and generally found to be suitable for solid waste testing.

4.4.1 In cases of doubt, contact the filter manufacturer to determine if the membrane or the prefilter is adversely affected by the particular waste. If no information is available, submerge the filter in the waste's liquid phase. After 48 hr, a filter that undergoes visible physical change (i.e., curls, dissolves, shrinks, or swells) is unsuitable for use.

4.4.2.1 Prepare a standard solution of the chemical species of interest.

4.4.2.2 Analyze the standard for its concentration of the chemical species.

4.4.2.3 Filter the standard and reanalyze. If the concentration of the filtrate differs from the original standard, the filter membrane leaches or absorbs one or more of the chemical species.

4.5 Structural integrity tester: One having a 3.18-cm-diameter hammer weighing 0.33 kg and having a free fall of 15.24 cm shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure A-4.

TABLE A-3. EPA-APPROVED FILTRATION MEDIA

Supplier	Filter to be used for aqueous systems	Filter to be used for organic systems
<u>Coarse prefilter</u>		
Gelman	61631, 61635	61631, 61635
Nuclepore	210907, 211707	210907, 211707
Millipore	AP25 035 00, AP25 127 50	AP25 035 00, AP25 127 50
<u>Medium prefilters</u>		
Nuclepore	210905, 211705	210905, 211705
Millipore	AP20 035 00, AP20 124 50	AP20 035 00, AP20 124 50
<u>Fine prefilters</u>		
Gelman	64798, 64803	64798, 64803
Nuclepore	210903, 211703	210903, 211703
Millipore	AP15 035 00, AP15 124 50	AP15 035 00, AP15 124 50
<u>Fine filters (0.45-?)</u>		
Gelman	60173, 60177	60540 or 66149, 60544 or 66151
Pall	NX04750, NX14225	
Nuclepore	142218	142218*
Millipore	HAWP 047 00, HAWP 142 50	FHUP 047 00, FHL P 142 50
Selas	83485-02, 83486-02	83485-02, 83486-02

\* Susceptible to decomposition by certain polar organic solvents.

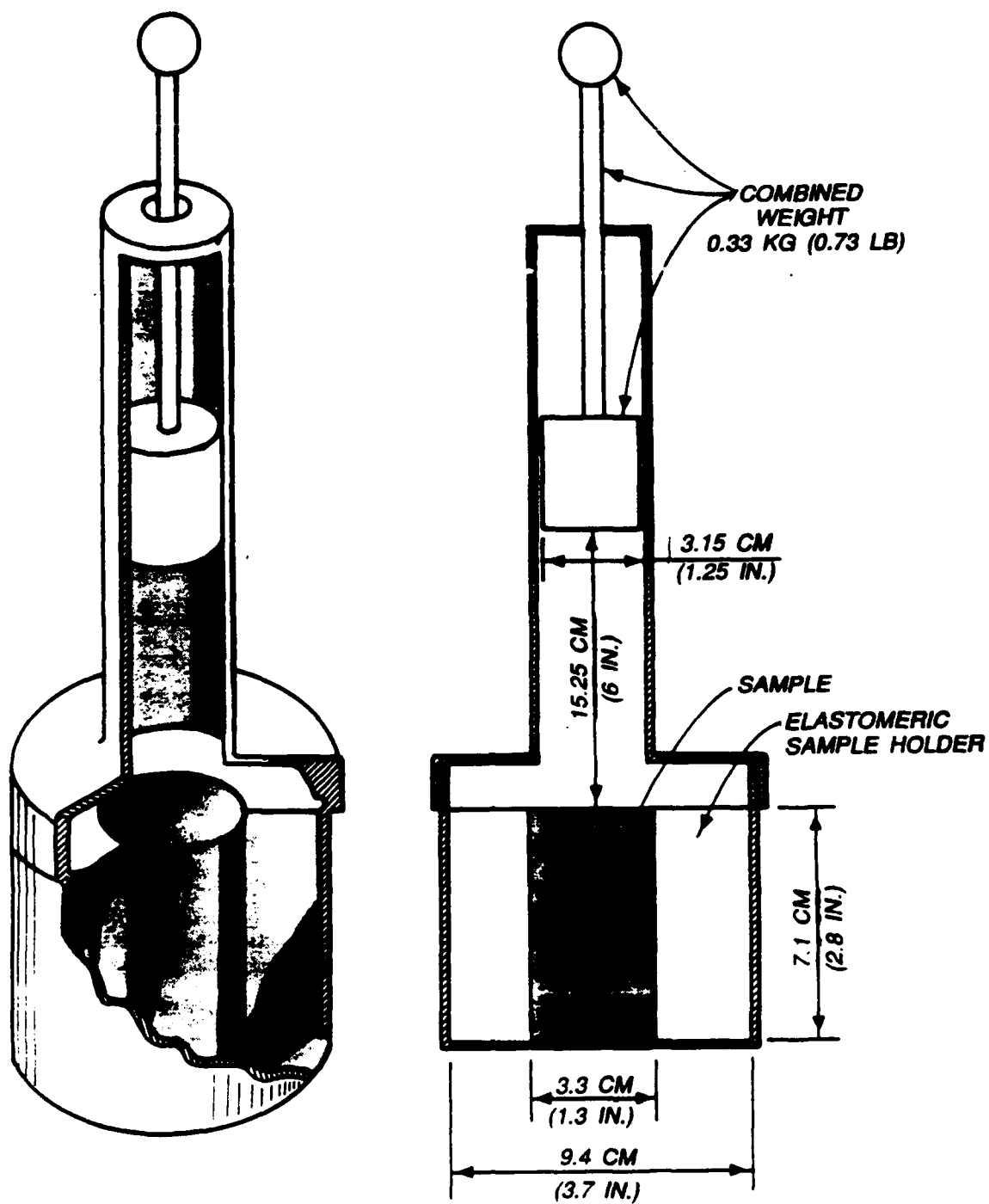


Figure A-4. EP compaction tester.

## 5.0 Reagents

5.1 Deionized water: Water should be monitored for impurities.

5.2 0.5 N acetic acid: This can be made by diluting concentrated glacial acetic acid (17.5 N). The glacial acetic acid should be of high purity and monitored for impurities.

5.3 Analytical standards should be prepared according to the analytical methods referenced in Table A-1.

## 6.0 Sample Collection, Preservation and Handling

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Section One of USEPA's SW-846.

6.2 Preservatives must not be added to samples.

6.3 Samples can be refrigerated if it is determined that refrigeration will not affect the integrity of the sample.

## 7.0 Procedure

7.1 If the waste does not contain any free liquid, go to Section 7.9. If the sample is liquid or multiphase, continue as follows. Weigh filter membrane and prefilter to  $\pm 0.01$  g. Handle membrane and prefilters with blunt curved-tip forceps or vacuum tweezers, or by applying suction with a pipette.

7.2 Assemble filter holder, membranes, and prefilters following the manufacturer's instructions. Place the 0.45- $\mu$ m membrane on the support screen and add prefilters in ascending order of pore size. Do not prewet filter membrane.

7.3 Weigh out a representative subsample of the waste (100 g minimum).

7.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.5 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10 to 15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops. This liquid will constitute part or all of the extract (refer to Section 7.16). The liquid should be refrigerated until time of analysis.

NOTE: Oil samples or samples that contain oil are treated in exactly the same way as any other sample. The liquid portion of the sample is filtered and treated as part of the EP extract. If the liquid portion of the sample will not filter (this is usually the case with heavy oils or greases), it is carried through the EP extraction as a solid.

7.6 Remove the solid phase and filter media and, while not allowing it to dry, weigh to  $\pm 0.01$  g. The wet weight of the residue is determined by calculating the weight difference between the weight of the filters (Section 7.1) and the weight of the solid phase and the filter media.

7.7 The waste will be handled differently from this point on depending on whether it contains more or less than 0.5% solids. If the sample appears to have less than 0.5% solids, the percent solids will be determined by the following procedure.

7.7.1 Dry the filter and residue at 80° C until two successive weighings yield the same value.

7.7.2 Calculate the percent solids using the following equation:

$$\frac{\text{Weight of filtered solid and filters} - \text{Tared weight of filters}}{\text{Initial weight of waste material}} \times 100 = \% \text{ solids}$$

NOTE: This procedure is only used to determine whether the solid must be extracted or whether it can be discarded unextracted. It is not used in calculating the amount of water or acid to use in the extraction step. Do not extract solid material that has been dried at 80° C. A new sample will have to be used for extraction if a percent solids determination is performed.

7.8 If the solid comprises less than 0.5% of the waste, discard the solid and proceed immediately to Section 7.17, treating the liquid phase as the extract.

7.9 The solid material obtained from Section 7.5 and all materials that do not contain free liquids should be evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than 3.1 cm<sup>2</sup> or passes through a 9.5-mm standard sieve, the operator should proceed to Section 7.11. If the surface area is smaller or the particle size larger than specified above, the solid material would be prepared for extraction by crushing, cutting, or grinding the material so that it passes through a 9.5-mm sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described in Section 7.10.

7.10 Structural Integrity Procedure (SIP):

7.10.1 Cut a 3.3-cm-diameter by 7.1-cm-long cylinder from the waste material! For wastes that have been treated using a fixation process, the waste may be cast in the form of a cylinder and allowed to cure for 30 days prior to testing.

7.10.2 Place waste into sample holder and assemble the tester. Raise the hammer to its maximum height and drop. Repeat 14 additional times.

7.10.3 Remove solid material from tester and scrape off any particles adhering to sample holder. Weigh the waste to the nearest 0.01 g and transfer it to the extractor.

7.11 If the sample contains more than 0.5% solids, use the wet weight of the solid phase obtained in Section 7.6 for purposes of calculating the amount of liquid and acid to employ for extraction by using the following equation:

$$W = W_f - W_t$$

where

$W$  - wet weight in grams of solid to be charged to extractor

$W_f$  - wet weight in grams of filtered solids and filter media

$W_t$  - weight in grams of tared filters

If the waste does not contain any free liquids, 100 g of the material will be subjected to the extraction procedure.

7.12 Place the appropriate amount of material (refer to Section 7.11) into the extractor and add 16 times its weight of deionized water.

7.13 After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 5.0, the pH of the solution should be decreased to  $5.0 \pm 0.2$  by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and, if the pH rises above 5.2, 0.5 N acetic acid should be added to lower the pH to  $5.0 \pm 0.2$ . However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hr and maintained at 20° to 40° C during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, OR, or its equivalent, in conjunction with a metering pump and reservoir of 0.5 N acetic acid. If such a system is not available, the following manual procedure shall be employed.

7.13.1 A pH meter should be calibrated in accordance with the manufacturer's specifications.

7.13.2 The pH of the solution should be checked and, if necessary, 0.5 N acetic acid should be manually added to the extractor until the pH reaches  $5.0 \pm 0.2$ . The pH of the solution should be adjusted at 15-, 30-, and 60-min intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5 pH unit.

7.13.3 The adjustment procedure should be continued for at least 6 hr.

7.13.4 If, at the end of the 24-hr extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to  $5.0 \pm 0.2$  and the extraction continued for an additional 4 hr, during which the pH should be adjusted at 1-hr intervals.

7.14 At the end of the extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

$$V = (20)(W) - 16(W) - A$$

where

V - milliliters of deionized water to be added

W - weight of solid, in grams, charged to extractor

A - milliliters of 0.5 N acetic acid added during extraction

7.15 The material in the extractor should be separated into its component liquid and solid phases in the following manner.

7.15.1 Allow slurries to stand to permit the solid phase to settle (wastes that are slow to settle may be centrifuged prior to filtration) and set up the filter apparatus (refer to Sections 4.3 and 4.4).

7.15.2 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10 to 15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops.

7.16 The liquids resulting from Sections 7.5 and 7.15 should be combined. This combined liquid (or the waste itself if it has less than 0.5% solids, as noted in Section 7.8) is the extract and should be analyzed for the presence of any of the contaminants specified in Table A-1 using the analytical procedures designated in Section 7.17.

7.17 The extract will be prepared and analyzed according to the analytical methods specified in Table A-1. All of these analytical methods are included in this manual. The method of standard addition will be employed for all metal analyses.

NOTE: If the EP extract includes two phases, concentration of contaminants is determined by using a simple weighted average. For example: An EP extract contains 50 ml of oil and 1,000 ml of an aqueous phase. Contaminant concentrations are determined for each phase. The final contamination concentration is taken to be

$$\frac{(50)(\text{Contaminant conc. in oil})}{1,050} + \frac{(1,000)(\text{Contaminant conc. of aqueous phase})}{1,050}$$

7.18 The extract concentrations are compared to the maximum contamination limits listed in Table A-1. If the extract concentrations are equal to or greater than the respective values, the waste is considered to be EP toxic.\*

#### 8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 All quality control measures suggested in the referenced analytical methods should be followed.

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\* Chromium concentrations have to be interpreted differently. A waste containing chromium will be determined to be EP toxic if (1) the waste extract has an initial pH of less than 7 and contains more than 5 mg/l of hexavalent chromium in the resulting extract, (2) the waste extract has an initial pH greater than 7 and a final pH greater than 7 and contains more than 5 mg/l of hexavalent chromium in the extract, or (3) the waste extract has an initial pH greater than 7 and a final pH less than 7 and contains more than 5 mg/l of total chromium, unless the chromium is trivalent. To determine whether the chromium is trivalent, the sample must be processed according to an alkaline digestion method (Method 3060) and analyzed for hexavalent chromium (Method 7195, 7196, or 7197).

## APPENDIX B

### TOXICITY CHARACTERISTIC LEACHING PROCEDURE\*

#### 1.0 Scope and Application

1.1 The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual contaminants are not present in the waste, or that they are present, but at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, the TCLP need not be run.

#### 2.0 Summary of Method (see Figure B-1)

2.1 For wastes containing less than 0.5% solids, the waste, after filtration through a 0.6- to 0.8- $\mu$  glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than 0.5% solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced (if necessary), weighed, and extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatiles (see Table B-1). Following extraction, the liquid extract is separated from the solid phase by 0.6- to 0.8- $\mu$  glass fiber filter filtration.

2.3 If compatible (e.g. precipitate or multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these liquids are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield the volume-weighted average concentration.

#### 3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

#### 4.0 Apparatus and Materials

4.1 Agitation apparatus: An acceptable agitation apparatus is one that is capable of rotating the extraction vessel in an end-over-end fashion (see Figure B-2) at  $30 \pm 2$  rpm. Suitable devices known to EPA are identified in Table B-2.

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\* Source: U.S. Environmental Protection Agency, 1986, Federal Register, Vol 51 (13 Jun), No. 114, Washington, DC.

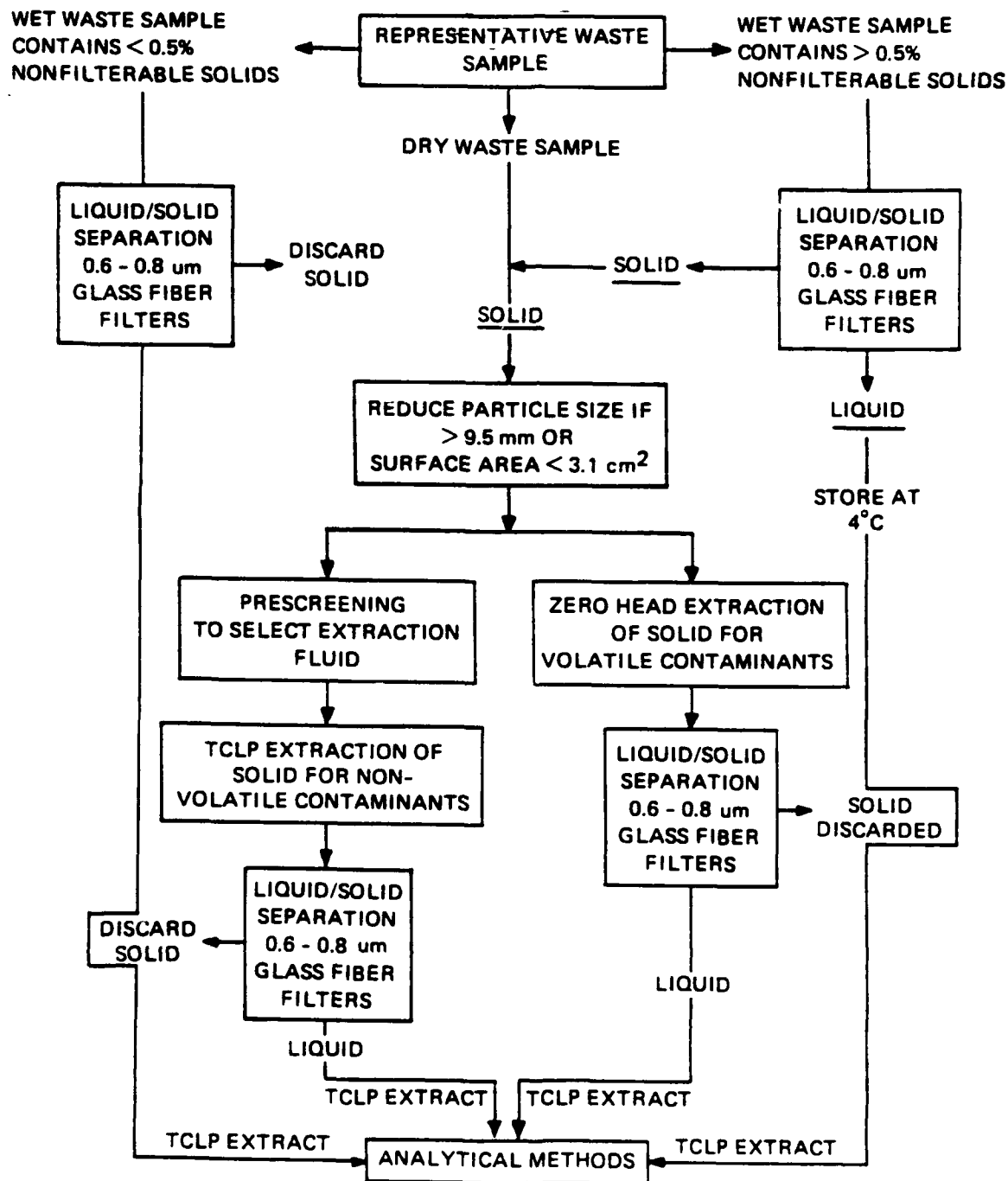


Figure B-1. TCLP flowchart.

TABLE B-1. VOLATILE CONTAMINANTS\*

Compound	
Acetone	67-64-1
Acrylonitrile	107-13-1
Benzene	71-43-2
n-Butyl alcohol	71-36-6
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethylene	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylane	1330-20-7

\* Includes compounds identified in both the Land Disposal Restrictions Rule and the Toxicity Characteristics.

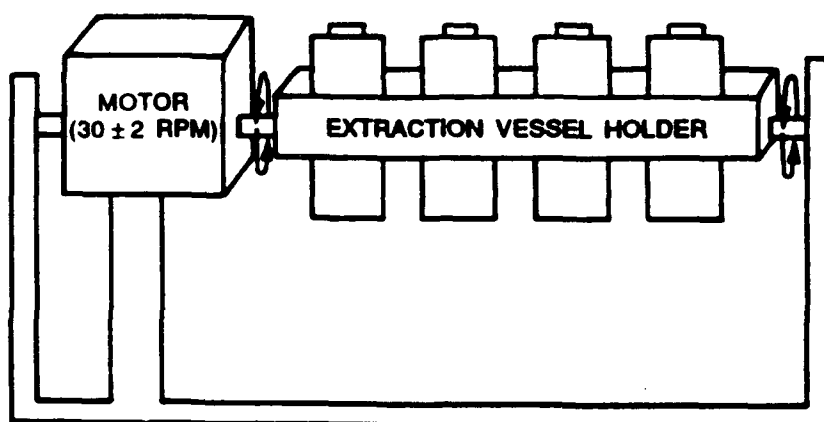


Figure B-2. TCLP rotary agitator.

TABLE B-2. SUITABLE ROTARY AGITATION APPARATUS\*

Company	Location	Model
Associated Design and Manufacturing Co.	Alexandria, Virginia (703)549-5999	4-vessel device 6-vessel device
Lars Lande Manufacturing	Whitmore Lake, Michigan (313)449-4116	10-vessel device
IRA Machine Shop and Laboratory	Santurce, Puerto Rico (809) 752-4004	16-vessel device
EPRI Extractor		6-vessel device

\* Any device which rotates the extraction vessel in an end-over-end fashion at  $30 \pm 2$  rpm is acceptable.

† Although this device is suitable, it is not commercially made. It may also require retrofitting to accommodate ZHE devices.

#### 4.2 Extraction vessel:

4.2.1 Zero-headspace extraction vessel (ZHE): When the waste is being tested for mobility of any volatile contaminants (see Table B-1), an extraction vessel which allows for liquid/solid separation within the device and which effectively precludes headspace (as depicted in Figure B-3) is used. This type of vessel allows for initial liquid/solid separation extraction and final extract filtration without having to open the vessel (see Section 4.3.1). These vessels shall have an internal volume of 500 to 600 ml and

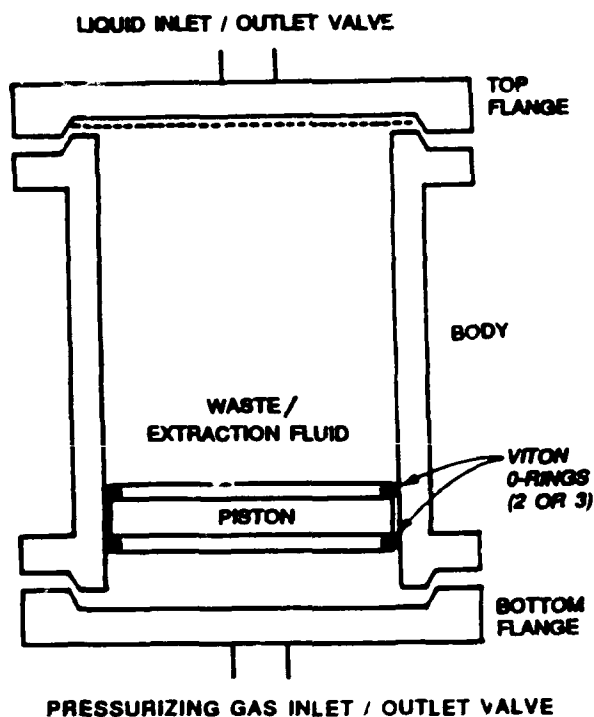


Figure B-3. TCLP zero-headspace extraction vessel.

be equipped to accommodate a 90-mm filter. Suitable ZHE devices known to EPA are identified in Table B-3. These devices contain viton O-rings which should be replaced frequently.

4.2.2 Other extraction vessels: When the waste is being evaluated for other than volatile contaminants, an extraction vessel that does not preclude headspace (e.g., 2-liter bottle) is used. Suitable extraction vessels include bottles made from various materials depending on the contaminants to be analyzed and the nature of the waste (see Section 4.3.3). These bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid-solid separation and final extract filtration.

TABLE B-3. SUITABLE ZERO-HEADSPACE EXTRACTOR VESSELS

Company	Location	Model No.
Associated Design and Manufacturing Co.	Alexandria, Virginia (703)549-5999	3740-ZHB
Millipore Corporation	Bedford, Massachusetts (800)225-3384	SD1P581C5

#### 4.3 Filtration devices:

4.3.1 Zero-headspace extractor vessel (see Figure B3): When the waste is being evaluated for volatiles, the zero-headspace extraction vessel is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the extract.

4.3.2 Filter holder: When the waste is being evaluated for other than volatile compounds, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation is used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressure up to 50 psi and more. The type of filter holder used depends on the properties of the material to be filtered (see Section 4.3.3). These devices shall have a minimum internal volume of 300 ml and be equipped to accommodate a minimum filter size of 47 mm. Filter holders known to EPA to be suitable for use are shown in Table B-4.

4.3.3 Materials of construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass polytetrafluoroethylene (PTFE) or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene, or polyvinyl chloride may be used when evaluating the mobility of metals.

4.4 Filters: Filters shall be made of borosilicate glass fiber, contain no binder materials, and have an effective pore size of 0.6 to 0.8  $\mu$  or equivalent. Filters known to EPA to meet these specifications are identified in Table B-5. Prefilters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1.0 N nitric acid followed by three consecutive rinses with deionized distilled water (minimum of 500 ml per rinse). Glass fiber filters are fragile and should be handled with care.

4.5 pH meters: Any of the commonly available pH meters are acceptable.

4.6 ZHE extract collection devices: Tedlar\* bags or glass, stainless steel, or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device.

4.7 ZHE extraction fluid collection devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g. a constant displacement pump, a gas-tight syringe, pressure filtration unit (see Section 4.3.2), or another ZHE device).

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\* Registered trademark of DuPont.

TABLE B-4. SUITABLE FILTER HOLDERS\*

Company	Location	Model	Size (mm)
Nuclepore Corporation	Pleasanton, California (800)882-7711	425910	142
		410400	47
Micro Filtration Systems	Dublin, California (415)828-6010	302400	142
Millipore Corporation	Bedford, Massachusetts (800)225-3384	YT30142HW	142
		XX1004700	47

\* Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic contaminants are of concern.

TABLE B-5. SUITABLE FILTER MEDIA

Company	Location	Model	Nominal Pore Size
Whatman Laboratory Products, Inc.	Clifton, New Jersey (201)773-5800	GFF	0.7

4.8 Laboratory balance: Any laboratory balance accurate to within  $\pm 0.01$  gram (g) may be used (all weight measurements are to be within  $\pm 0.1$  g).

#### 5.0 Reagents

5.1 Water: ASTM Type 1 deionized, carbon treated, decarbonized, filtered water (or equivalent water that is treated to remove volatile components) shall be used when evaluating wastes for volatile contaminants. Otherwise, ASTM Type 2 deionized distilled water (or equivalent) is used. These waters should be monitored periodically for impurities.

5.2 1.0 N Hydrochloric acid (HCl) made from ACS Reagent grade.

5.3 1.0 N Nitric acid (HNO<sub>3</sub>) made from ACS Reagent grade.

5.4 1.0 N Sodium hydroxide (NaOH) made from ACS Reagent grade.

5.5 Glacial acetic acid (HOAc) made from ACS Reagent grade.

5.6 Extraction fluid:

5.6.1 Extraction fluid 1: This fluid is made by adding 5.7 ml glacial HOAc to 500 ml of the appropriate water (see Section 5.1), adding 64.3 ml of 1.0 N NaOH, and diluting to a volume of 1 liter. When correctly prepared, the pH of this fluid will be  $4.93 \pm 0.05$ .

5.6.2 Extraction fluid 2: This fluid is made by diluting 5.7 ml glacial HOAc with ASTM Type 2 water (see Section 5.1) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be  $2.88 \pm 0.05$ .

NOTE: These extraction fluids shall be made up fresh daily. The pH should be checked prior to use to ensure that the fluids are made up accurately, and they should be monitored frequently for impurities.

5.7 Analytical standards shall be prepared according to the appropriate analytical method.

## 6.0 Sample Collection, Preservation, and Handling

6.1 All samples shall be collected using a sampling plan that addresses the considerations discussed in "Test Methods for Evaluating Solid Wastes" (SW-846).

6.2 Preservatives shall not be added to samples.

6.3 Samples can be refrigerated unless it results in irreversible physical changes to the waste.

6.4 When the waste is to be evaluated for volatile contaminants, care must be taken to ensure that these are not lost. Samples shall be taken and stored in a manner which prevents the loss of volatile contaminants. If possible, any necessary particle size reduction should be conducted as the sample is being taken (see Step 8.5). Refer to SW-846 for additional sampling and storage requirements when volatiles are contaminants of concern.

6.5 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. If they need to be stored, even for a short period of time, storage shall be at 4° C, and samples for volatiles analysis shall not be allowed to come into contact with the atmosphere (i.e., no headspace).

## 7.0 Procedure When Volatiles Are Not Involved

NOTES: Although a minimum sample size of 100 g is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough waste sample should be collected such that at least 75 g of the solid phase of the waste (as determined using glass fiber filter filtration) is extracted. This will ensure that there is adequate extract for the required analyses (e.g. semivolatiles, metals, pesticides, and herbicides).

The determination of which extraction fluid to use (see Step 7.12) may also be conducted at the start of this procedure. This determination shall be based on the solid phase of the waste (as obtained using glass fiber filter filtration).

7.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample of the waste (100-g minimum) and proceed to Step 7.11.

7.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device discussed in Section 4.3.2 and outlined in Steps 7.3 to 7.9.

7.3 Preweigh the filter and the container that will receive the filtrate.

7.4 Assemble filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter if evaluating the mobility of metals (see Section 4.4).

7.5 Weigh out a representative subsample of the waste (100-g minimum) and record weight.

7.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.7 Transfer the waste sample to the filter holder.

NOTES: If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.5, to determine the weight of the waste sample that will be filtered. Gradually apply vacuum or gentle pressure of 1 to 10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-min interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter and no additional liquid has passed through the filter in any 2-min interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., does not result in any additional filtrate within any 2-min period), filtration is stopped.

Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid; however, even after applying vacuum or pressure filtration as outlined in Step 7.7, this material may not filter. If this is the case, the material within

the filtration device is defined as a solid and is carried through the extraction as a solid.

7.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Step 7.3) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (see Step 7.15) or stored at 4° C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Step 7.5 or 7.7. Record the weight of the liquid and solid phases.

NOTE: If the weight of the solid phase of the waste is less than 75 g, review Step 7.0.

7.10 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% solids. If the sample obviously has greater than 0.5% solids, go to Step 7.11. If it appears that the solid may comprise less than 0.5% of the total waste, the percent solids will be determined as follows:

7.10.1 Remove the solid phase and filter from the filtration apparatus.

7.10.2 Dry the filter and solid phase at  $100 \pm 20^\circ$  C until two successive weighings yield the same value. Record final weight.

7.10.3 Calculate the percent solids as follows: Weight of dry waste and filters minus tared weight of filters divided by initial weight of waste (Step 7.5 or 7.7) multiplied by 100 equals percent solids.

7.10.4 If the solid comprises less than 0.5% of the waste, the solid is discarded, and the liquid phase is defined as the TCLP extract. Proceed to Step 7.14.

7.10.5 If the solid is greater than or equal to 0.5% of the waste, return to Step 7.1, and begin the procedure with a new sample of waste. Do not extract the solid that has been dried.

NOTE: This step is only used to determine whether the solid must be extracted or whether it may be discarded unextracted. It is not used in calculating the amount of extraction fluid to use in extracting the waste, nor is the dried solid that is derived from this step subjected to extraction. A new sample will have to be prepared for extraction.

7.11 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than  $3.1 \text{ cm}^2$  or is capable of passing through a 9.5-mm standard sieve, proceed to Step 7.12. If the surface area is smaller or the particle size is larger than that described above, the solid material is prepared for extraction by crushing, cutting, or grinding the solid material to a surface area or particle size as described above. When surface area or particle size has been appropriately altered, proceed to Step 7.12.

7.12 This step describes the determination of the appropriate extracting fluid to use (see Sections 5.0 and 7.0).

7.12.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0-g portion to a 500-ml beaker or Erlenmeyer flask.

7.12.2 Add 96.5 ml distilled deionized water (ASTM Type 2), cover with watchglass, and stir vigorously for 5 min using a magnetic stirrer. Measure and record the pH. If the pH is  $<5.0$ , extraction fluid 1 is used. Proceed to Step 7.13.

7.12.3 If the pH from Step 7.12.2 is  $>5.0$ , add 3.5 ml 1.0 N HCl, slurry for 30 sec, cover with a watchglass, heat to  $50^{\circ}\text{C}$ , and hold for 10 min.

7.12.4 Let the solution cool to room temperature and record pH. If pH is  $<5.0$ , use extraction fluid 1. If the pH is  $>5.0$ , extraction fluid 2 is used.

7.13 Calculate the weight of the remaining solid material by subtracting the weight of the subsample taken for Step 7.12 from the original amount of solid material, as obtained from Step 7.1 or 7.9. Transfer remaining solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

NOTES: If any of the solid phase remains adhered to the walls of the filter holder, or the container used to transfer the waste, its weight shall be determined and subtracted from the weight of the solid phase of the waste, as determined above; this weight is used in calculating the amount of extraction fluid to add into the extractor bottle.

Slowly add an amount of the appropriate extraction fluid (see Step 7.12) into the extractor bottle equal to 20 times the weight of the solid phase that has been placed into the extractor bottle. Close extractor bottle tightly, secure in rotary extractor device, and rotate at  $30 \pm 2$  rpm for 18 hr. The temperature shall be maintained at  $22^{\circ} \pm 3^{\circ}\text{C}$  during the extraction period.

As agitation continues, pressure may build up within the extractor bottle (due to the evolution of gases such as carbon dioxide). To relieve these pressures, the extractor bottle may be periodically opened and vented into a hood.

7.14 Following the 18-hr extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter as outlined in Step 7.7. This new filter shall be acid-washed (see Section 4.4) if evaluating the mobility of metals.

7.15 The TCLP extract is now prepared as follows:

7.15.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 7.14 is defined as the TCLP extract. Proceed to Step 7.16.

7.15.2 If compatible (e.g. will not form precipitate or multiple phases), the filtered liquid resulting from Step 7.14 is combined with the initial liquid phase of the waste as obtained in Step 7.9. This combined liquid is defined as the TCLP extract. Proceed to Step 7.16.

7.15.3 If the initial liquid phase of the waste, as obtained from Step 7.9, is not or may not be compatible with the filtered liquid resulting from Step 7.14, these liquids are not combined. These liquids are collectively defined as the TCLP extract, analyzed separately, and the results combined mathematically. Proceed to Step 7.16.

7.16 The TCLP extract will be prepared and analyzed according to the appropriate SW-846 analytical methods identified in Appendix III of 40 CFR 261. TCLP extracts to be analyzed for metals shall be acid-digested. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to 0.1 ml), conduct the appropriate analyses, and combine the results mathematically by using a simple weighted average:

$$\text{Final contaminant concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where

$V_1$  = volume of the first phase, liters

$C_1$  = concentration of the contaminant of concern in the first phase, milligrams per liter

$V_2$  = volume of the second phase, liters

$C_2$  = concentration of the contaminant of concern in the second phase, milligrams per liter

7.17 The contaminant concentrations in the TCLP extract are compared to the thresholds identified in the appropriate regulations. Refer to Section 9 for quality assurance requirements.

## 8.0 Procedure When Volatiles Are Involved

NOTES: The ZHE device has approximately a 500-ml internal capacity. Although a minimum sample size of 100 g was required in the Section 7 procedure, the ZHE can only accommodate a maximum 100-percent solids sample of 25 g, due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Step 8.4 provides the means by which to determine the approximate sample size for the ZHE device.

Although the following procedure allows for particle size reduction during the conduct of the procedure, this could result in the loss of volatile compounds. If possible, any necessary particle size reduction (see Step 8.5) should be conducted on the sample as it is being taken.

Particle size reduction should only be conducted during the procedure if there is no other choice.

In carrying out the following steps, do not allow the waste to be exposed to the atmosphere for any more time than is absolutely necessary.

8.1 Preweigh the (evacuated) container that will receive the filtrate (see Section 4.6), and set aside.

8.2 Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston O-rings slightly with extraction fluid). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

8.3 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample of the waste (25-g maximum - see Step 8.0), record weight, and proceed to Step 8.5.

8.4 This step provides the means by which to determine the approximate sample size for the ZHE device. If the waste is liquid or multiphasic, follow the procedure outlined in Steps 7.2 to 7.9 (using the Section 7 filtration apparatus) and obtain the percent solids by dividing the weight of the solid phase of the waste by the original sample size used. If the waste obviously contains greater than 0.5% solids, go to Step 8.4.2. If it appears that the solid may comprise less than 0.5% of the waste, go to Step 8.4.1.

8.4.1 Determine the percent solids by using the procedure outlined in Step 7.10. If the waste contains less than 0.5% solids, weigh out a new 100-g minimum representative sample, proceed to Step 8.7, and follow until the liquid phase of the waste is filtered using the ZHE device (Step 8.8). This liquid filtrate is defined as the TCLP extract and is analyzed directly. If the waste contains greater than or equal to 0.5% solids, repeat Step 8.4 using a new 100-g minimum sample, determine the percent solids, and proceed to Step 8.4.2.

8.4.2 If the sample is <25% solids, weigh out a new 100-g minimum representative sample and proceed to Step 8.5. If the sample is >25% solids, the maximum amount of sample the ZHE can accommodate is determined by dividing 25 g by the percent solids obtained from Step 8.4. Weigh out a new representative sample of the determined size.

8.5 After a representative sample of the waste (sample size determined from Step 8.4) has been weighed out and recorded, the sample is now evaluated for particle size (see Step 8.0). If the solid material within the waste obviously has a surface area per gram of material equal to or greater than  $3.1 \text{ cm}^2$ , or is capable of passing through a 9.5-mm standard sieve, proceed immediately to Step 8.6. If the surface area is smaller or the particle size is larger than that described above, the solid material that does not meet the above criteria is separated from the liquid phase by sieving (or equivalent means), and the solid is prepared for extraction by crushing, cutting, or grinding to a surface area or particle size as described above.

NOTE: Wastes and appropriate equipment should be refrigerated, if possible, to 4° C prior to particle size reduction. Grinding and milling machinery which generates heat shall not be used for particle size reduction. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible. When surface area or particle size has been appropriately altered, the solid is recombined with the rest of the waste.

8.6 Waste slurries need not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration.

8.7 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 8.4, to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange), and with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1 to 10 psi (or more if necessary) to slowly force all headspace out of the ZHE device. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

8.8 Attach evacuated preweighed filtrate collection container to the liquid inlet/outlet valve and open valve. Begin applying gentle pressure of 1 to 10 psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any 2-min interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2-min interval, proceed to the next 10-psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within any 2-min period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.9 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid; however, even after applying pressure filtration, this material will not filter.

If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained less than 0.5% solids (see Step 8.4), this filtrate is defined as the TCLP extraction and is analyzed directly. Proceed to Step 8.13.

8.10 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Step 8.1) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (see Steps 8.13 and 8.14) or stored at 4° C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample (see Step 8.4). Record the final weight of the liquid and solid phases.

8.11 The following paragraphs detail the addition of the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid 1 is used in all cases (see Section 5.6).

8.11.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the amount of fluid introduced into the device equals 20 times the weight of the solid phase of the waste that is in the ZHE.

8.11.2. After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to make sure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Put 5 to 10 psi behind the piston (if necessary), and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Repressurize the ZHE with 5 to 10 psi and check all ZHE fittings to ensure that they are closed.

8.11.3 Place the ZHE in the rotary extractor apparatus (if it is not already there) and rotate the ZHE at  $30 \pm 2$  rpm for 18 hr. The temperature shall be maintained at  $22^\circ \pm 3^\circ$  C during agitation.

8.12 Following the 18-hr extraction, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Replace ZHE O-rings or other fittings, as necessary, and redo the extraction with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e. Tedlar bag, gas-tight syringe) holding the initial liquid phase of the waste, unless doing so would create multiple phases or there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter,

using the ZHE device as discussed in Step 8.8. All extract shall be filtered and collected if the extract is multiphasic or if the waste contained an initial liquid phase.

NOTE: If the glass fiber filter is not intact following agitation, the filtration device discussed in the Note to Section 4.3.1 may be used to filter the material within the ZHE.

8.13 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 8.12 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Step 8.12 and the initial liquid phase (Step 8.8) are collectively defined as the TCLP extract.

8.14 The TCLP extract will be prepared and analyzed according to the appropriate SW-846 analytical methods, as identified in Appendix III of 40 CFR 261. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to 0.1 ml), conduct the appropriate analyses, and combine the results mathematically by using a simple volume weighted average:

$$\text{Final contaminant concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where

$V_1$  - volume of the first phase, liters

$C_1$  - concentration of the contaminant of concern in the first phase, milligrams per liter

$V_2$  - volume of the second phase, liters

$C_2$  - concentration of the contaminant of concern in the second phase, milligrams per liter

8.15 The contaminant concentrations in the TCLP extract are compared to the thresholds identified in the appropriate regulations. Refer to Section 9 for quality assurance requirements.

## 9.0 Quality Assurance Requirements

9.1 All data, including quality assurance data, should be maintained and available for reference or inspection.

9.2 A minimum of one blank for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment are occurring. One blank shall also be employed for every new batch of leaching fluid that is made up.

9.3 All quality control measures described in the appropriate analytical methods shall be followed.

9.4 The method of standard addition shall be employed for each waste type if recovery of the compound from spiked splits of the TCLP extract is not between 50% and 150% or if the concentration of the constituent measured in the extract is within 20% of the appropriate regulatory threshold. If more than one extraction is being run on samples of the same waste, the method of

standard addition need only be applied once and the percent recoveries applied to the remainder of the extractions.

9.5 TCLP extracts shall be analyzed within the following periods after generation: volatiles - 14 days; semivolatiles - 40 days; mercury - 28 days; other metals - 180 days.

## APPENDIX C

### LABORATORY DETERMINATION OF MOISTURE CONTENT OF HAZARDOUS WASTE MATERIALS

#### BACKGROUND

This method was developed to determine the moisture content of raw and solidified/stabilized hazardous waste materials. Due to the wide diversity of properties which hazardous wastes may exhibit, this method cannot address, nor is it applicable to, all waste types. Caution must be utilized when applying this method. It may be necessary to modify this method to address conditions mandated by the waste. ASTM method D 2216-80 was utilized as a guide in preparing this method.

#### SIGNIFICANCE AND USE

The waste content of a material is defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid materials particles. A hazardous waste material may contain various constituents which may artificially add or subtract from the results of moisture content. Such variables include: (1) chemically bound water (water of hydration) which may be released at relative low temperature, thus appearing as free water loss, (2) organic materials which oxidize at low temperature, and (3) any condition, except for "free" water loss, which may increase or decrease the weight of sample upon drying. Discretion must be utilized when applying this method to ensure such situations are considered and steps are taken to provide results consistent with the purpose of the test.

#### APPARATUS

Drying oven - thermostatically controlled, preferably of the forced-draft type, and capable of maintaining a uniform temperature of 60° C in the drying chamber. This oven should also be capable of maintaining approximately 110° C. If a forced-draft oven is used, the draft should not be strong enough to "blow" any sample from the specimen container.

Balances - having a precision of  $\pm 0.0001$  g.

Specimen containers - suitable containers made of materials resistant to corrosion and a change in mass upon repeated heating and cooling.

Mortar and pestle - capable of reducing the particle size of the waste to 2.0 mm or less.

Sieve - a 2.0-mm (No. 10) sieve.

Desiccator - a desiccator of suitable size containing a hydrous compound.

## SAMPLES

In all cases, representative portions of the material being sampled should be collected. To ensure representative sampling, a great deal of thought and planning will be necessary prior to any sampling activities. The USEPA has suggested sampling procedures as outlined in "Test Methods for Evaluating Solid Waste," SW-846, 2nd ed. Following sample collection, large samples should be ground and homogenized prior to collecting the subsample. The moisture determination should be performed as soon as possible after the subsample has been collected.

## PROCEDURE

1. Select a representative subsample in accordance with the previous section.
2. Place the undried sample in a clean dry mortar and grind the sample to pass a No. 10 sieve. Approximately 30 g of sample should be sieved and rehomogenized in an appropriate dry container. Note: The moisture determination should be performed on the ground sample as soon as possible; if the sample must be stored for any period of time, it should be placed in a dry, labeled, sealed container having minimal headspace.
3. Dry each sample container in the oven at 110° C and cool to room temperature prior to performing Step 4.
4. Using tongs to transfer the sample containers, weigh 3 dry labeled sample containers and record their weights ( $W_c$ ). Tongs should be used in all subsequent sample transfers. Do not touch the sample containers, except with the tongs, once they have been dried.
5. Divide the sieved sample into three equal portions and place approximately 10 g of the moist sieved sample in each of the containers from Step 4. Reweigh each container and record its weight ( $W_w$ ). Care should be taken to avoid spilling any of the sample material; if any spillage occurs, this sample should be discarded.
6. Place each sample in the drying oven maintained at a temperature of  $60^\circ \pm 3^\circ$  C. Dry each sample for a minimum period of 6 hr.
7. At the end of the 6-hr period, remove the sample container containing the largest mass of sample and place it in the desiccator. Allow the sample to reach room temperature in the desiccator; then weigh this sample and record its weight ( $W_{d1}$ ,  $W_{d2}$ , etc.).
8. Replace the sample used in Step 7 back in the oven and dry for a minimum of an additional hour. Repeat Step 7 until this sample reaches a constant weight ( $W_d$ ). Note: Constant weight for this procedure is defined as a mass change of less than 0.1% of the total sample weight between two successive drying periods of a minimum of 1 hr. After this sample has reached a constant weight, repeat Step 7 for the remaining samples.

## CALCULATIONS

Calculate the constant weight as follows:

$$W_{\text{cat}} = \{ [W_{d(i-1)} - W_{d(i)}] / W_{d(i)} \} * 100 \quad (\text{C-1})$$

where

$W_{\text{cat}}$  = constant weight of the largest sample expressed as a percentage  
( $W_{\text{cat}}$  must be less than 0.1%)

$W_{d(i-1)}$  = weight of the largest sample, one weighing before the final constant weight was taken, g

$W_{d(i)}$  = weight of the largest sample at the final constant weight, g

$$M_r = [W_w - W_{d(i)}] / [W_w - W_c] \quad (\text{C-2})$$

where

$M_r$  = moisture content expressed as a percentage

$W_w$  = weight of the undried sample, g

$W_c$  = weight of the dried sample container, g

$$M_a = (M_{r1} + M_{r2} + M_{r3}) / 3 \quad (\text{C-3})$$

where

$M_a$  = average moisture content expressed as a percentage

$M_{r1}, M_{r2}, M_{r3}$  = moisture content of each sample

## QUALITY CONTROL/QUALITY ASSURANCE

The following calculation is utilized to calculate the percent deviation ( $P_d$ ):

$$P_d = (M_{r1} - M_a) / M_a * 100$$

The percent deviation is calculated for each sample. If the percent deviation is greater than 2%, these data are discarded, and a complete moisture analysis is repeated.

## REPORT

The report (data sheet) shall include the following:

1. Identification of the sample being tested, by sample number.

2. Water content of the specimen, which is an average of three specimens.
3. Any unusual characteristic of the sample that should be noted.
4. Any deviation from this protocol.

## APPENDIX D

## PHYSICAL PROPERTIES OF THE ORGANIC COMPOUNDS

TABLE D-1. PHYSICAL PROPERTIES OF ORGANIC COMPOUNDS USED IN THIS STUDY

Compound	Molecular Weight	Vapor Pressure* (mm Hg)	Solubility* (mg/l)	Boiling Point (C)
Benzene	78.11	95.2	820-1,800	80.1
2-Butanone	72.10	77.5	353,000 <sup>+</sup>	79.6
Carbon disulfide	76.14	260*	2,300 <sup>++</sup>	46.3
Carbon tetrachloride	153.82	90.0	785	76.54
Chlorobenzene	112.56	8.8	500	132
Chloroform	119.38	150.5	8,200	61.7
1,2 Dichloroethane	98.98	61.0	8,690	83.47
1,1 Dichloroethene	96.94	591.0**	400	37.0
Ethylbenzene	106.16	5.0	152	136.2
4-Methyl-2-Pentanone	100.20	6.0	17,000	116-159
1,1,2,2 Tetrachloroethane	167.86	5.0	2,900	146.4
Tetrachloroethene	165.83	14.0	150-200	121.0
1,1,1 Trichloroethane	133.41	96.0	480-4,400	74.1
1,1,2 Trichloroethane	133.41	19.0	4,500	113.7
Trichloroethene	131.39	57.9	1,100	87.0
Toluene	92.10	28.7**	234.8**	110.8

Source- All values except those named below were taken from "Water-Related Environmental Fate of 129 Priority Pollutants; Volume II" (USEPA 1979).

Values for 2-Butanone, 4-Methyl-2-Pentanone, 1,1,2 Trichloroethane, chlorobenzene, and carbon disulfide were taken from Handbook of Environmental Data on Organic Chemicals (Verschuere 1977)

- \* Values reported at 20° C.
- \*\* Values reported at 25° C.
- + Value reported at 10° C.
- ++ Value reported at 22° C.

# APPENDIX E

## STUDY A RAW DATA

TABLE E-1. TCLP AND EP EXTRACT ANALYSIS FOR CADMIUM

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid*/ Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Oil	0%	EP	R1	400	0.0208	0.000255
			R2	400	0.0206	0.000252
		TCLP	R1	II	0.0073	0.000089
			R2	II	0.0015	0.000018
	2%	EP	R1	400	0.0033	0.000042
			R2	400	0.0034	0.000041
		TCLP	R1	II	0.0015	0.000018
			P2	II	0.0015	0.000018
	5%	EP	R1	400	0.002	0.00002
			R2	400	0.0015	0.000018
		TCLP	R1	II	0.0188	0.000230
			R2	II	0.0092	0.00011
	8%	EP	R1	400	0.0031	0.000037
			R2	400	0.0041	0.000048
		TCLP	R1	II	0.0138	0.000163
			R2	II	0.0021	0.000025
Grease	0%	EP	R1	400	<0.0001	0.000001
			R2	400	0.0171	0.000205
		TCLP	R1	II	0.0002	0.000002
			R2	II	0.0007	0.000008
	2%	EP	R1	400	0.004	0.00005
			R2	400	0.0168	0.000198
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001
	5%	EP	R1	400	0.0022	0.000026
			R2	400	0.0094	0.00011
		TCLP	R1	II	<0.0001	0.000001
			R2	II	0.0003	0.000004
	8%	EP	R1	400	0.0072	0.000084
			R2	400	0.0135	0.000157
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001

(Continued)

\* II - TCLP extraction fluid 2.

(Sheet 1 of 5)

TABLE E-1 (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Lead	0%	EP	R1	400	0.0027	0.000027
			R2	400	0.0013	0.000013
		TCLP	R1	II	0.0023	0.000023
			R2	II	0.001	0.00001
	2%	EP	R1	400	0.0051	0.000051
			R2	400	0.0042	0.000042
	2%	TCLP	R1	II	0.0057	0.000057
			R2	II	0.0133	0.000133
	5%	EP	R1	400	0.0093	0.000093
			R2	400	0.0013	0.000013
		TCLP	R1	II	0.096	0.00096
			R2	II	0.089	0.00089
	8%	EP	R1	400	0.074	0.00074
			R2	400	0.0139	0.000139
		TCLP	R1	II	0.015	0.00015
			R2	II	0.0298	0.000298
Copper	0%	EP	R1	400	0.0039	0.000039
			R2	400	0.0016	0.000016
		TCLP	R1	II	0.0001	0.000001
			R2	II	<0.0001	0.000001
	2%	EP	R1	400	0.0002	0.000002
			R2	400	0.0053	0.000052
		TCLP	R1	II	0.0006	0.000006
			R2	II	0.0005	0.000005
	5%	EP	R1	400	0.0029	0.000029
			R2	400	0.0037	0.000037
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001
	8%	EP	R1	400	0.0021	0.000021
			R2	400	0.0011	0.000011
		TCLP	R1	II	0.0008	0.000008
			R2	II	<0.0001	0.000001
Zinc	0%	EP	R1	400	0.0956	0.000956
			R2	400	<0.0001	0.000001
		TCLP	R1	II	<0.0001	0.000001
			R2	II	0.011	0.000110
	2%	EP	R1	400	0.0077	0.000077
			R2	400	0.0081	0.000081
		TCLP	R1	II	0.0014	0.000016
			R2	II	0.0015	0.000015

(Continued)

(Sheet 2 of 5)

TABLE E-1 (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Zinc (Cont.)	5%	EP	R1	400	0.0036	0.000036
			R2	400	0.0087	0.000087
		TCLP	R1	II	0.0048	0.000054
			R2	II	0.0039	0.000039
	8%	EP	R1	400	0.0046	0.000046
			R2	400	0.0023	0.000023
		TCLP	R1	II	0.0017	0.000017
			R2	II	0.003	0.000034
Hexachloro-benzene	0%	EP	R1	400	0.0416	0.000498
			R2	400	0.0037	0.000044
		TCLP	R1	II	0.0027	0.000032
			R2	II	0.0028	0.000034
	2%	EP	R1	400	0.006	0.000073
			R2	400	0.0072	0.000088
		TCLP	R1	II	0.0025	0.000031
			R2	II	0.0018	0.000022
	5%	EP	R1	400	0.0378	0.000463
			R2	400	0.0056	0.000067
		TCLP	R1	II	0.0167	0.000205
			R2	II	0.0088	0.00011
	8%	EP	R1	400	0.0042	0.000052
			R2	400	0.0066	0.000081
		TCLP	R1	II	0.0003	0.000004
			R2	II	0.0001	0.000001
Trichloro-ethene	0%	EP	R1	400	0.0015	0.000018
			R2	400	0.0013	0.000015
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001
	2%	EP	R1	400	0.0014	0.000017
			R2	400	0.0015	0.000018
		TCLP	R1	II	0.0006	0.000007
			R2	II	<0.0001	0.000001
	5%	EP	R1	400	0.0005	0.000006
			R2	400	0.0022	0.000026
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001
	8%	EP	R1	400	0.001	0.00001
			R2	400	0.0004	0.000005
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001

(Continued)

(Sheet 3 of 5)

TABLE E-1 (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Phenol	0%	EP	R1	400	0.0006	0.000007
			R2	400	0.0024	0.000029
	2%	TCLP	R1	II	0.0012	0.000015
			R2	II	0.0028	0.000034
		EP	R1	400	0.0014	0.000017
			R2	400	0.0028	0.000035
		TCLP	R1	II	0.0022	0.000027
			R2	II	0.0061	0.000076
	5%	EP	R1	400	0.0067	0.000083
			R2	400	0.0026	0.000032
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001
	8%	EP	R1	400	0.0048	0.000059
			R2	400	0.0026	0.000032
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001
Sodium sulfate	0%	EP	R1	400	0.0071	0.000084
			R2	400	0.0058	0.000069
		TCLP	R1	II	0.0008	0.000009
			R2	II	0.0018	0.000021
	2%	EP	R1	400	0.0057	0.000068
			R2	400	0.0091	0.00011
		TCLP	R1	II	0.0009	0.00001
			R2	II	0.0009	0.00001
	5%	EP	R1	400	0.002	0.00002
			R2	400	0.0115	0.000139
		TCLP	R1	II	0.0009	0.00001
			R2	II	0.0010	0.00001
	8%	EP	R1	400	0.0106	0.000129
			R2	400	0.0063	0.000077
		TCLP	R1	II	0.0015	0.000018
			R2	II	0.0025	0.000030
Sodium hydroxide	0%	EP	R1	400	<0.0001	0.000001
			R2	400	0.0011	0.000012
		TCLP	R1	II	0.0004	0.000004
			R2	II	<0.0001	0.000001
	2%	EP	R1	400	0.0045	0.000053
			R2	400	0.0061	0.000071
		TCLP	R1	II	0.0002	0.000002
			R2	II	0.0004	0.000005

(Continued)

(Sheet 4 of 5)

TABLE E-1 (Concluded)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Sodium hydroxide (Cont.)	5%	EP	R1	400	0.0034	0.000042
			R2	400	0.0027	0.000034
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001
	8%	EP	R1	400	0.0024	0.000028
			R2	400	0.0016	0.000019
		TCLP	R1	II	<0.0001	0.000001
			R2	II	<0.0001	0.000001

TABLE E-2. TCLP AND EP EXTRACT ANALYSIS FOR CHROMIUM

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid*/ Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Oil	0%	EP	R1	400	0.044	0.00054
			R2	400	0.036	0.00044
		TCLP	R1	II	0.147	0.00180
			R2	II	0.037	0.00045
	2%	EP	R1	400	0.015	0.00018
			R2	400	0.019	0.00023
		TCLP	R1	II	0.007	0.00009
			R2	II	0.114	0.00141
	5%	EP	R1	400	0.036	0.00044
			R2	400	0.024	0.00029
		TCLP	R1	II	0.007	0.00009
			R2	II	0.030	0.0004
	8%	EP	R1	400	0.004	0.00004
			R2	400	0.030	0.0003
		TCLP	R1	II	0.046	0.00054
			R2	II	0.044	0.00052
Grease	0%	EP	R1	400	0.284	0.00340
			R2	400	0.209	0.00250
		TCLP	R1	II	0.049	0.00059
			R2	II	0.039	0.00047
	2%	EP	R1	400	0.061	0.00072
			R2	400	0.043	0.00051
		TCLP	R1	II	0.012	0.00014
			R2	II	0.01	0.0001
	5%	EP	R1	400	0.062	0.00073
			R2	400	0.034	0.00040
		TCLP	R1	II	0.04	0.0005
			R2	II	0.017	0.00020
	8%	EP	R1	400	0.03	0.0003
			R2	400	0.036	0.00042
		TCLP	R1	II	0.014	0.00016
			R2	II	0.015	0.00017
Lead	0%	EP	R1	400	0.033	0.00033
			R2	400	0.065	0.00065
		TCLP	R1	II	0.046	0.00046
			R2	II	0.055	0.00055
	2%	EP	R1	400	0.044	0.00044
			R2	400	0.029	0.00029

(Continued)

\* II = TCLP extraction fluid 2.

(Sheet 1 of 4)

TABLE E-2 (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Lead (Cont.)	2%	TCLP	R1	II	0.058	0.00058
			R2	II	0.055	0.00055
	5%	EP	R1	400	0.031	0.00031
			R2	400	0.029	0.00029
		TCLP	R1	II	0.047	0.00047
			R2	II	0.04	0.00040
	8%	EP	R1	400	0.03	0.0003
			R2	400	0.03	0.0003
		TCLP	R1	II	0.038	0.00038
			R2	II	0.085	0.00085
Copper	0%	EP	R1	400	0.01	0.0001
			R2	400	0.009	0.00009
		TCLP	R1	II	0.038	0.00038
			R2	II	0.039	0.00039
	2%	EP	R1	400	0.114	0.00114
			R2	400	0.061	0.00061
		TCLP	R1	II	0.071	0.00071
			R2	II	0.065	0.00065
	5%	EP	R1	400	0.038	0.00038
			R2	400	0.034	0.00034
		TCLP	R1	II	0.048	0.00048
			R2	II	0.049	0.00049
	8%	EP	R1	400	0.011	0.00011
			R2	400	0.01	0.0001
		TCLP	R1	II	0.043	0.00043
			R2	II	0.049	0.00049
Zinc	0%	EP	R1	400	0.051	0.00051
			R2	400	0.019	0.00019
		TCLP	R1	II	0.071	0.00080
			R2	II	0.065	0.00065
	2%	EP	R1	400	0.047	0.00047
			R2	400	0.044	0.00044
		TCLP	R1	II	0.062	0.00070
			R2	II	0.062	0.00062
	5%	EP	R1	400	0.098	0.00098
			R2	400	0.07	0.0007
		TCLP	R1	II	0.1	0.001
			R2	II	0.096	0.00096
	8%	EP	R1	400	0.09	0.0009
			R2	400	0.101	0.00101
		TCLP	R1	II	0.095	0.00095
			R2	II	0.093	0.0010

(Continued)

(Sheet 2 of 4)

TABLE E-2 (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Hexachloro-benzene	0%	EP	R1	400	0.015	0.00018
			R2	400	0.041	0.00049
		TCLP	R1	II	0.251	0.00300
			R2	II	0.349	0.00418
	2%	EP	R1	400	0.07	0.0009
			R2	400	0.031	0.00038
		TCLP	R1	II	0.3	0.004
			R2	II	0.18	0.0022
	5%	EP	R1	400	0.008	0.0001
			R2	400	0.013	0.00016
		TCLP	R1	II	0.689	0.00844
			R2	II	0.332	0.00407
	8%	EP	R1	400	0.032	0.00039
			R2	400	0.089	0.0011
		TCLP	R1	II	0.041	0.00051
			R2	II	0.029	0.00036
Trichloro-ethene	0%	EP	R1	400	0.041	0.00049
			R2	400	0.04	0.0005
		TCLP	R1	II	0.076	0.00091
			R2	II	0.077	0.00092
	2%	EP	R1	400	0.046	0.00055
			R2	400	0.048	0.00057
		TCLP	R1	II	0.089	0.0011
			R2	II	0.076	0.00090
	5%	EP	R1	400	0.038	0.00046
			R2	400	0.035	0.00042
		TCLP	R1	II	0.072	0.00087
			R2	II	0.073	0.00088
	8%	EP	R1	400	0.037	0.00044
			R2	400	0.064	0.00077
		TCLP	R1	II	0.064	0.00077
			R2	II	0.066	0.00079
Phenol	0%	EP	R1	400	0.016	0.00020
			R2	400	0.013	0.00016
		TCLP	R1	II	0.072	0.00088
			R2	II	0.124	0.00152
	2%	EP	R1	400	0.007	0.00009
			R2	400	0.01	0.0001
		TCLP	R1	II	0.108	0.00134
			R2	II	0.216	0.00268

(Continued)

(Sheet 3 of 4)

TABLE E-2 (Concluded)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Phenol (Cont.)	5%	EP	R1	400	0.006	0.00007
			R2	400	0.005	0.00006
		TCLP	R1	II	0.050	0.0006
			R2	II	0.045	0.00056
	8%	EP	R1	400	0.02	0.0002
			R2	400	0.019	0.00023
		TCLP	R1	II	0.015	0.00019
			R2	II	0.016	0.00020
		EP	R1	400	0.077	0.00091
			R2	400	0.065	0.00077
Sodium sulfate	0%	TCLP	R1	II	0.049	0.00058
			R2	II	0.049	0.00058
	2%	EP	R1	400	0.122	0.00146
			R2	400	0.13	0.00156
		TCLP	R1	II	0.153	0.00183
			R2	II	0.156	0.00186
	5%	EP	R1	400	0.131	0.00183
			R2	400	0.155	0.00188
		TCLP	R1	II	0.143	0.00173
			R2	II	0.144	0.00174
	8%	EP	R1	400	0.14	0.0017
			R2	400	0.166	0.00202
		TCLP	R1	II	0.146	0.00178
			R2	II	0.144	0.00175
Sodium hydroxide	0%	EP	R1	400	0.07	0.0008
			R2	400	0.1	0.001
		TCLP	R1	II	0.08	0.0009
			R2	II	0.08	0.0009
	2%	EP	R1	400	0.126	0.00147
			R2	400	0.138	0.00161
		TCLP	R1	II	0.113	0.00132
			R2	II	0.118	0.00138
	5%	EP	R1	400	0.485	0.00603
			R2	400	0.483	0.00600
		TCLP	R1	II	0.411	0.00511
			R2	II	0.415	0.00516
	8%	EP	R1	400	0.377	0.00444
			R2	400	0.381	0.00449
		TCLP	R1	II	0.307	0.00362
			R2	II	0.327	0.00385

(Sheet 4 of 4)

TABLE E-3. TCLP AND EP EXTRACT ANALYSIS FOR MERCURY

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid*/ Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Oil	0%	EP	R1	400	0.4	0.005
			R2	400	0.388	0.00475
		TCLP	R1	II	0.453	0.00554
			R2	II	0.425	0.00520
	2%	EP	R1	400	0.0157	0.000194
			R2	400	0.0319	0.000393
		TCLP	R1	II	0.0287	0.000354
			R2	II	0.0276	0.000340
	5%	EP	R1	400	0.003	0.00003
			R2	400	0.0032	0.000039
		TCLP	R1	II	0.0046	0.000056
			R2	II	0.0068	0.000083
	8%	EP	R1	400	0.0011	0.000013
			R2	400	0.0011	0.000013
		TCLP	R1	II	0.0021	0.000025
			R2	II	0.0023	0.000027
Grease	0%	EP	R1	400	0.266	0.00319
			R2	400	0.243	0.00291
		TCLP	R1	II	0.249	0.00298
			R2	II	0.203	0.00243
	2%	EP	R1	400	0.092	0.0012
			R2	400	0.169	0.00199
		TCLP	R1	II	0.134	0.00158
			R2	II	0.157	0.00185
	5%	EP	R1	400	0.066	0.00077
			R2	400	0.132	0.00154
		TCLP	R1	II	0.103	0.00120
			R2	II	0.088	0.0010
	8%	EP	R1	400	0.069	0.00080
			R2	400	0.137	0.00159
		TCLP	R1	II	0.092	0.0011
			R2	II	0.106	0.00123
Lead	0%	EP	R1	400	0.437	0.00437
			R2	400	0.264	0.00264
		TCLP	R1	II	0.498	0.00498
			R2	II	0.494	0.50494
	2%	EP	R1	400	0.21	0.0021
			R2	400	0.276	0.00276

(Continued)

\* II = TCLP extraction fluid 2.

(Sheet 1 of 5)

TABLE E-3. (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Lead (Cont.)	2%	TCLP	R1	II	0.501	0.00501
			R2	II	0.498	0.00498
	5%	EP	R1	400	0.107	0.00107
			R2	400	0.284	0.00284
		TCLP	R1	II	0.37	0.0037
			R2	II	0.443	0.00443
	8%	EP	R1	400	0.291	0.00291
			R2	400	0.222	0.00222
		TCLP	R1	II	0.447	0.00447
			R2	II	0.49	0.0049
Copper	0%	EP	R1	400	0.17	0.0017
			R2	400	0.148	0.00148
		TCLP	R1	II	0.287	0.00287
			R2	II	0.225	0.00225
	2%	EP	R1	400	0.164	0.00164
			R2	400	0.357	0.00357
		TCLP	R1	II	0.287	0.00287
			R2	II	0.332	0.00332
	5%	EP	R1	400	0.346	0.00346
			R2	400	0.353	0.00353
		TCLP	R1	II	0.205	0.00205
			R2	II	0.285	0.00285
	8%	EP	R1	400	0.195	0.00195
			R2	400	0.234	0.00234
		TCLP	R1	II	0.24	0.0024
			R2	II	0.264	0.00264
Zinc	0%	EP	R1	400	0.191	0.00191
			R2	400	0.193	0.00193
		TCLP	R1	II	0.32	0.0036
			R2	II	0.285	0.00285
	2%	EP	R1	400	0.274	0.00274
			R2	400	0.285	0.00285
		TCLP	R1	II	0.279	0.00313
			R2	II	0.282	0.00282
	5%	EP	R1	400	0.158	0.00158
			R2	400	0.231	0.00231
		TCLP	R1	II	0.26	0.0029
			R2	II	0.263	0.00263
	8%	EP	R1	400	0.142	0.00142
			R2	400	0.097	0.00097

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TABLE E-3. (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Zinc (Cont.)	8%	TCLP	R1	II	0.249	0.00249
			R2	II	0.22	0.0025
Hexachloro-benzene	0%	EP	R1		0.2830	0.00338792
			R2		0.2340	0.00280132
		TCLP	R1	II	0.241	0.00289
			R2	II	0.322	0.00385
	2%	EP	R1	400	0.238	0.00290
			R2	400	0.287	0.00350
		TCLP	R1	II	0.276	0.00337
			R2	II	0.269	0.00328
	5%	EP	R1	400	0.234	0.00287
			R2	400	0.227	0.00278
		TCLP	R1	II	0.318	0.00390
			R2	II	0.245	0.00300
	8%	EP	R1	400	0.269	0.00332
			R2	400	0.217	0.00268
		TCLP	R1	II	0.206	0.00254
			R2	II	0.245	0.00302
Trichloro-ethene	0%	EP	R1	400	0.419	0.00500
			R2	400	0.425	0.00507
		TCLP	R1	II	0.271	0.00323
			R2	II	0.184	0.00220
	2%	EP	R1	400	0.375	0.00445
			R2	400	0.384	0.00456
		TCLP	R1	II	0.248	0.00295
			R2	II	0.305	0.00362
	5%	EP	R1	400	0.641	0.00771
			R2	400	0.602	0.00724
		TCLP	R1	II	0.392	0.00471
			R2	II	0.456	0.00548
	8%	EP	R1	400	0.69	0.0083
			R2	400	0.1	0.001
		TCLP	R1	II	0.697	0.00837
			R2	II	0.643	0.00772
Phenol	0%	EP	R1	400	0.381	0.00468
			R2	400	0.282	0.00346
		TCLP	R1	II	0.356	0.00437
			R2	II	0.381	0.00468

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(Sheet 3 of 5)

TABLE E-3. (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Phenol (Cont.)	2%	EP	R1	400	1.3	0.016
			R2	400	1.32	0.0164
		TCLP	R1	II	1.3	0.016
			R2	II	1.3	0.016
	5%	EP	R1	400	1.3	0.016
			R2	400	1.3	0.016
		TCLP	R1	II	1.35	0.0178
			R2	II	1.3	0.016
	8%	EP	R1	400	1.32	0.0163
			R2	400	1.32	0.0163
		TCLP	R1	II	1.48	0.0183
			R2	II	1.35	0.0167
Sodium sulfate	0%	EP	R1	400	0.199	0.00236
			R2	400	0.141	0.00167
		TCLP	R1	II	0.257	0.00305
			R2	II	0.246	0.00292
	2%	EP	R1	400	0.166	0.00198
			R2	400	0.124	0.00148
		TCLP	R1	II	0.304	0.00363
			R2	II	0.257	0.00307
	5%	EP	R1	400	0.094	0.0011
			R2	400	0.135	0.00163
		TCLP	R1	II	0.226	0.00273
			R2	II	0.185	0.00224
	8%	EP	R1	400	0.152	0.00185
			R2	400	0.16	0.0019
		TCLP	R1	II	0.199	0.00242
			R2	II	0.21	0.0026
Sodium hydroxide	0%	EP	R1	400	0.18	0.0020
			R2	400	0.111	0.00125
		TCLP	R1	II	0.151	0.00170
			R2	II	0.155	0.00174
	2%	EP	R1	400	0.29	0.0034
			R2	400	0.264	0.00308
		TCLP	R1	II	0.198	0.00231
			R2	II	0.195	0.00228
	5%	EP	R1	400	0.272	0.00338
			R2	400	0.347	0.00431
		TCLP	R1	II	0.193	0.00240
			R2	II	0.186	0.00231

(Continued)

(Sheet 4 of 5)

TABLE E-3. (Concluded)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Sodium hydroxide (Cont.)	8%	EP	R1	400	0.326	0.00384
			R2	400	0.249	0.00293
		TCLP	R1	II	0.199	0.00234
			R2	II	0.376	0.00443

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TABLE E-4. TCLP AND EP EXTRACTS FOR NICKEL

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid*/ Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Oil	0%	EP	R1	400	0.07	0.0009
			R2	400	0.068	0.00083
		TCLP	R1	II	0.068	0.00083
			R2	II	0.03	0.0004
	2%	EP	R1	400	0.067	0.00083
			R2	400	0.068	0.00084
		TCLP	R1	II	0.001	0.00001
			R2	II	0.074	0.00091
	5%	EP	R1	400	0.066	0.00081
			R2	400	0.065	0.00080
		TCLP	R1	II	0.014	0.00017
			R2	II	0.011	0.00013
	8%	EP	R1	400	0.063	0.00074
			R2	400	0.066	0.00078
		TCLP	R1	II	0.053	0.00062
			R2	II	0.092	0.0011
Grease	0%	EP	R1	400	0.201	0.00241
			R2	400	0.154	0.00184
		TCLP	R1	II	0.012	0.00014
			R2	II	0.041	0.00049
	2%	EP	R1	400	0.014	0.00016
			R2	400	0.02	0.0002
		TCLP	R1	II	0.015	0.00018
			R2	II	0.014	0.00016
	5%	EP	R1	400	0.006	0.00007
			R2	400	0.006	0.00007
		TCLP	R1	II	0.009	0.0001
			R2	II	0.014	0.00016
	8%	EP	R1	400	0.002	0.00002
			R2	400	0.015	0.00017
		TCLP	R1	II	0.01	0.0001
			R2	II	0.007	0.00008
Lead	0%	EP	R1	400	0.013	0.00013
			R2	400	0.012	0.00012
		TCLP	R1	II	0.031	0.00031
			R2	II	0.031	0.00031

(Continued)

\* II = TCLP extraction fluid 2.

(Sheet 1 of 5)

TABLE E-4. (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Lead (Cont.)	2%	EP	R1	400	0.005	0.00005
			R2	400	0.012	0.00012
		TCLP	R1	II	0.015	0.00015
			R2	II	0.015	0.00015
	5%	EP	R1	400	0.009	0.00009
			R2	400	0.006	0.00006
		TCLP	R1	II	0.052	0.00052
			R2	II	0.043	0.00043
	8%	EP	R1	400	0.026	0.00026
			R2	400	0.024	0.00024
		TCLP	R1	II	0.073	0.00073
			R2	II	0.117	0.00117
Copper	0%	EP	R1	400	0.012	0.00012
			R2	400	0.022	0.00022
		TCLP	R1	II	0.03	0.00030
			R2	II	0.013	0.00013
	2%	EP	R1	400	0.014	0.00014
			R2	400	0.033	0.00033
		TCLP	R1	II	0.035	0.00035
			R2	II	0.022	0.00022
	5%	EP	R1	400	0.011	0.00011
			R2	400	0.018	0.00018
		TCLP	R1	II	0.044	0.00044
			R2	II	0.012	0.00012
	8%	EP	R1	400	0.018	0.00018
			R2	400	0.017	0.00017
		TCLP	R1	II	0.079	0.00079
			R2	II	0.024	0.00024
Zinc	0%	EP	R1	400	0.066	0.00066
			R2	400	0.064	0.00064
		TCLP	R1	II	0.036	0.00040
			R2	II	0.03	0.00030
	2%	EP	R1	400	0.084	0.00084
			R2	400	0.098	0.00098
		TCLP	R1	II	0.0006	0.000007
			R2	II	0.006	0.00006
	5%	EP	R1	400	0.089	0.00089
			R2	400	0.095	0.00095
		TCLP	R1	II	0.011	0.00012
			R2	II	0.011	0.00011

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(Sheet 2 of 5)

TABLE E-4. (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Zinc (Cont.)	8%	EP	R1	400	0.107	0.00107
			R2	400	0.1	0.001
		TCLP	R1	II	0.13	0.0013
			R2	II	0.007	0.00008
Hexachloro-benzene	0%	EP	R1	400	0.01	0.0001
			R2	400	0.029	0.00035
		TCLP	R1	II	0.074	0.00089
			R2	II	0.102	0.00122
	2%	EP	R1	400	0.011	0.00010
			R2	400	0.018	0.00022
		TCLP	R1	II	0.075	0.00095
			R2	II	0.043	0.00052
	5%	EP	R1	400	0.015	0.00018
			R2	400	0.013	0.00016
		TCLP	R1	II	0.203	0.00249
			R2	II	0.1	0.001
	8%	EP	R1	400	0.017	0.00021
			R2	400	0.278	0.00343
		TCLP	R1	II	0.027	0.00033
			R2	II	0.021	0.00025
Trichloro-ethene	0%	EP	R1	400	0.012	0.00014
			R2	400	0.017	0.00020
		TCLP	R1	II	0.003	0.00004
			R2	II	0.006	0.00007
	2%	EP	R1	400	0.009	0.0001
			R2	400	0.01	0.0001
		TCLP	R1	II	0.012	0.00014
			R2	II	0.003	0.00004
	5%	EP	R1	400	0.009	0.0001
			R2	400	0.013	0.00016
		TCLP	R1	II	0.02	0.0002
			R2	II	0.003	0.00004
	8%	EP	R1	400	0.008	0.0001
			R2	400	0.012	0.00014
		TCLP	R1	II	0.005	0.00006
			R2	II	0.003	0.00004
Phenol	0%	EP	R1	400	0.012	0.00015
			R2	400	0.006	0.00007

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TABLE E-4. (Continued)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Phenol (Cont.)	0%	TCLP	R1	II	0.061	0.00075
			R2	II	0.091	0.0011
	2%	EP	R1	400	0.005	0.00006
			R2	400	0.005	0.00006
		TCLP	R1	II	0.058	0.00072
			R2	II	0.1	0.001
	5%	EP	R1	400	0.004	0.00005
			R2	400	0.011	0.00014
		TCLP	R1	II	0.035	0.00044
			R2	II	0.037	0.00046
	8%	EP	R1	400	0.002	0.00002
			R2	400	0.007	0.00009
		TCLP	R1	II	0.033	0.00041
			R2	II	0.046	0.00057
Sodium sulfate	0%	EP	R1	400	0.063	0.00075
			R2	400	0.06	0.0007
		TCLP	R1	II	0.102	0.00121
			R2	II	0.087	0.0010
	2%	EP	R1	400	0.057	0.00068
			R2	400	0.059	0.00070
		TCLP	R1	II	0.069	0.00082
			R2	II	0.071	0.00085
	5%	EP	R1	400	0.057	0.00069
			R2	400	0.073	0.00088
		TCLP	R1	II	0.063	0.00076
			R2	II	0.063	0.00076
	8%	EP	R1	400	0.066	0.00080
			R2	400	0.059	0.00072
		TCLP	R1	II	0.056	0.00068
			R2	II	0.055	0.00067
Sodium hydroxide	0%	EP	R1	400	0.086	0.00097
			R2	400	0.073	0.00082
		TCLP	R1	II	0.065	0.00073
			R2	II	0.065	0.00073
	2%	EP	R1	400	0.017	0.00020
			R2	400	0.102	0.00119
		TCLP	R1	II	0.054	0.00063
			R2	II	0.056	0.00065

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(Sheet 4 of 5)

TABLE E-4. (Concluded)

Interference Compound	Interference Concentration	Extraction Test	Replicate	Extraction Fluid/Acid Added (ml)	Extracted Concentration (mg/l)	Normalized Extraction Concentration, (mg/kg)
Sodium hydroxide (Cont.)	5%	EP	R1	400	0.008	0.0001
			R2	400	0.006	0.00007
		TCLP	R1	II	0.07	0.0009
			R2	II	0.064	0.00080
	8%	EP	R1	400	0.003	0.00004
			R2	400	0.001	0.00001
		TCLP	R1	II	0.04	0.0004
			R2	II	0.033	0.00039

(Sheet 5 of 5)

## APPENDIX F

### GRAPHICAL REPRESENTATION OF THE RESULTS OF TCLP AND EP EXTRACTIONS FOR STUDY A METALS

Figures F1-F4 are graphical representations of the TCLP and EP extractions for each metal contaminant of Study A. In these figures the normalized EP extract concentrations are plotted versus the normalized TCLP extract concentrations. A line with a slope of 1.0 is plotted on each graph. Points which lie on this line indicate that the extract concentrations for the EP and TCLP are equal. Points above this line indicate that the TCLP produced extracts with higher concentrations of the contaminant, and points below this line indicate that the EP resulted in extracts containing higher concentrations of the contaminants. Based on this information, the mercury data (Figure F-4) indicate that the TCLP was the more aggressive extraction method because more than 70 percent of the mercury data points lie above the line.

In order to compare Figures F-1 through F-4, the difference in scales must be considered. The scales for the chromium and mercury data, presented in Figures F-2 and F-4, are equivalent. However, scales for the cadmium and nickel data, presented in Figures F-1 and F-3, cannot be adjusted to match the scales of Figures F-2 and F-4 and still maintain any reasonable resolution. Therefore, the scale for nickel is 2.8 times smaller and the scale for the cadmium data is 17 times smaller than those used in the other figures.

The data presented in Figures F-1 and F-3 are closely grouped near the line, indicating equal EP and TCLP extract concentrations. Comparison of Figures F-1 and F-3 to Figure F-4 illustrates that the results for the EP and TCLP extracts for mercury differ and that the EP and TCLP extracts for cadmium and nickel do not. Similar observations for the chromium are more difficult to decipher.

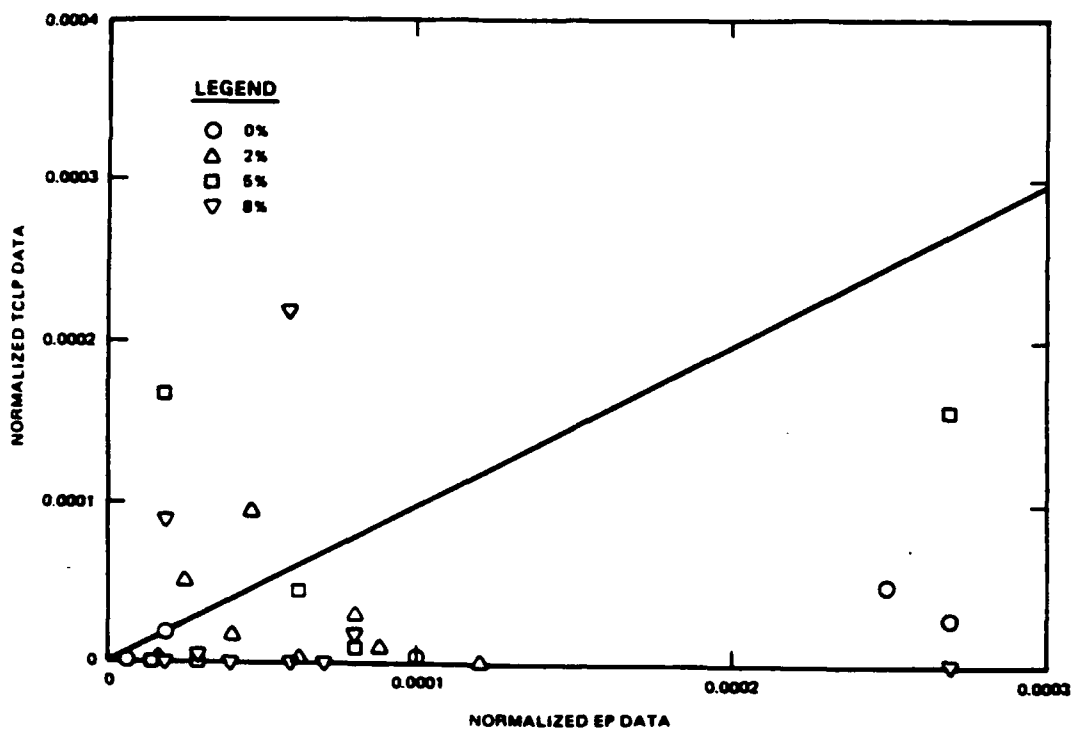


Figure F-1. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study A cadmium contaminant.

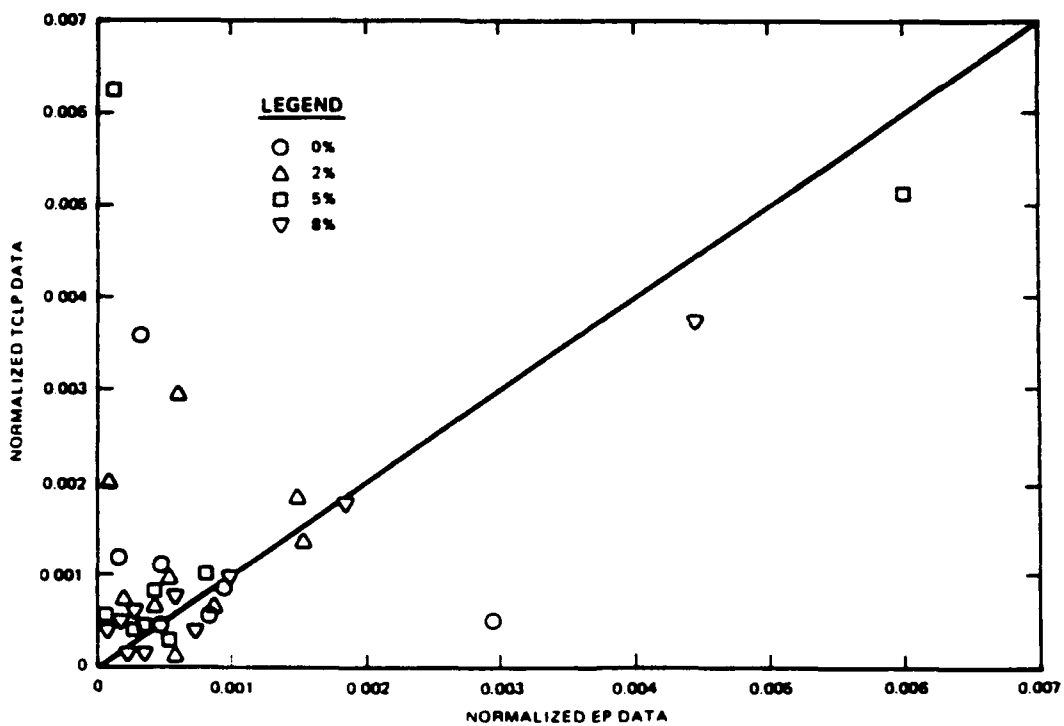


Figure F-2. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study A chromium contaminant.

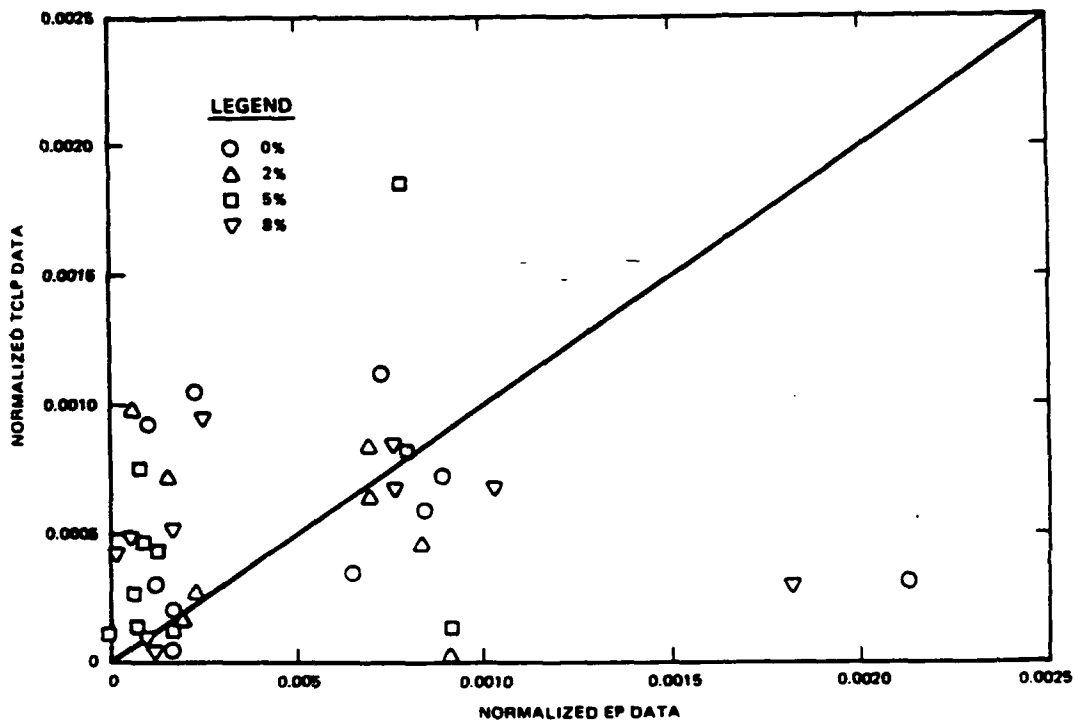


Figure F-3. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study A nickel contaminant.

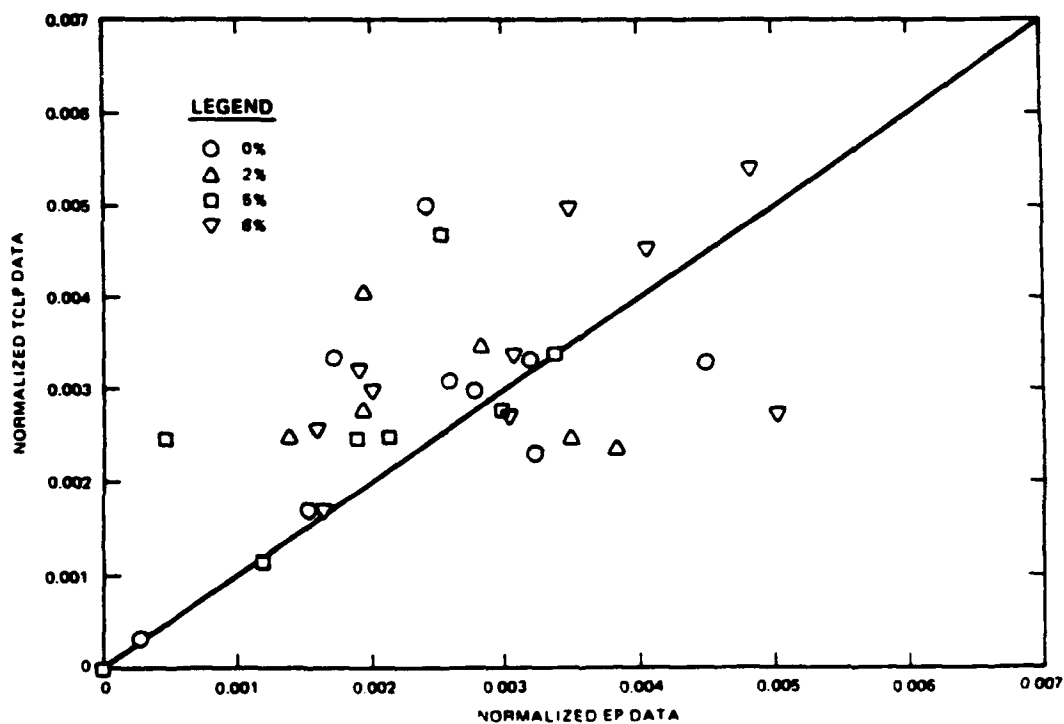


Figure F-4. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study A mercury contaminant.

## APPENDIX G

## STUDY B METALS RAW DATA

TABLE G-1. STUDY B TCLP AND EP EXTRACT ANALYSIS FOR THE WES SLUDGE METAL CONTAMINANTS

Metal Contaminant	Test	Organic Level	Replicate Number	Extraction Fluid*/ Acid Added (ml)	Extract Concentration (mg/l)	Normalized Extracted Concentration (mg/l)
Cadmium	EP	0.1%	R1	400	0.0012	0.000024
			R2	400	0.0013	0.000025
			R3	400	0.0016	0.000031
		1%	R1	400	0.0006	0.00001
			R2	400	0.06	0.001
			R3	400	0.0303	0.000608
	TCLP	0.1%	R1	II	0.0051	0.00010
			R2	II	0.0094	0.00018
			R3	II	0.0164	0.000322
		1%	R1	II	0.0074	0.00015
			R2	II	0.0073	0.00015
			R3	II	0.0057	0.00011
Chromium	EP	0.1%	R1	400	0.027	0.00053
			R2	400	0.023	0.00045
			R3	400	0.021	0.00041
		1%	R1	400	0.019	0.00038
			R2	400	0.313	0.00628
			R3	400	0.058	0.0012
	TCLP	0.1%	R1	II	0.049	0.00096
			R2	II	0.062	0.0012
			R3	II	0.099	0.0019
		1%	R1	II	0.065	0.0013
			R2	II	0.056	0.0011
			R3	II	0.048	0.00096
Nickel	EP	0.1%	R1	400	0.034	0.00067
			R2	400	0.011	0.00022
			R3	400	0.019	0.00037
		1%	R1	400	0.032	0.00064
			R2	400	0.352	0.00706
			R3	400	0.169	0.00339

(Continued)

\* II - TCLP extraction fluid 2.

TABLE G-1 (Concluded)

TABLE G-1 (Continued)								
Metal Contaminant	Test	Organic Level	Replicate Number	Extraction Fluid/Acid Added (ml)	Extract Concentration (mg/l)	Normalized Extracted Concentration (mg/l)		
Nickel (Cont.)	TCLP	0.1%	R1	II	0.11	0.0022		
			R2	II	0.154	0.00302		
			R3	II	0.096	0.0019		
		1%	R1	II	0.235	0.00471		
			R2	II	0.214	0.00429		
			R3	II	0.169	0.00335		
		Mercury	EP	0.1%	R1	400	8.48	0.166
					R2	400	7.57	0.148
					R3	400	7.82	0.153
1%	R1			400	0.01	0.0004		
	R2			400	0.02	0.0004		
	R3			400	0.01	0.0003		
TCLP	0.1%			R1	II	7.9	0.16	
				R2	II	7.9	0.15	
				R3	II	7.6	0.15	
	1%		R1	II	8.5	0.17		
			R2	II	8.3	0.17		
			R3	II	7.9	0.16		

TABLE G-2. STUDY B TCLP AND EP EXTRACT ANALYSIS FOR THE WTC WASTE  
METAL CONTAMINANTS

Metal Contaminant	Test	Organic Level	Replicate Number	Extraction Fluid*/ Acid Added (ml)	Extract Concentration (mg/l)	Normalized Extracted Concentration (mg/l)
Arsenic	EP	0.1%	R1	400	0.022	0.00027
			R2	400	0.019	0.00023
			R3	400	0.022	0.00027
		1%	R1	400	0.032	0.00040
			R2	400	0.027	0.00034
			R3	400	0.024	0.00030
	TCLP	0.1%	R1	II	0.058	0.00071
			R2	II	0.053	0.00065
			R3	II	0.053	0.00065
		1%	R1	II	0.104	0.0011
			R2	II	0.136	0.0017
			R3	II	0.122	0.00153
Cadmium	EP	0.1%	R1	400	0.005	6.E-6
			R2	400	<0.001	1.E-6
			R3	400	0.006	7.E-6
		1%	R1	400	<0.001	1.E-6
			R2	400	<0.001	1.E-6
			R3	400	<0.001	1.E-6
	TCLP	0.1%	R1	II	<0.001	1.E-6
			R2	II	0.002	2.E-6
			R3	II	0.004	5.E-6
		1%	R1	II	0.005	6.E-6
			R2	II	<0.001	1.E-6
			R3	II	<0.001	1.E-6
Chromium	EP	0.1%	R1	400	0.041	0.00050
			R2	400	0.041	0.00050
			R3	400	0.041	0.00050
		1%	R1	400	0.031	0.00039
			R2	400	0.035	0.00044
			R3	400	0.03	0.0004
	TCLP	0.1%	R1	II	0.049	0.00060
			R2	II	0.035	0.00043
			R3	II	0.035	0.00043

(Continued)

\* II = TCLP extraction fluid 2.

TABLE G-2 (Concluded)

Metal Contaminant	Test	Organic Level	Replicate Number	Extraction Fluid/Acid Added (ml)	Extract Concentration (mg/l)	Normalized Extracted Concentration (mg/l)
Chromium (Cont.)	TCLP	1%	R1	II	0.035	0.00044
			R2	II	0.036	0.00045
			R3	II	0.036	0.00045
Lead	EP	0.1%	R1	400	0.005	0.00006
			R2	400	0.009	0.0001
			R3	400	0.008	0.0001
		1%	R1	400	0.011	0.00014
			R2	400	0.013	0.00016
			R3	400	0.015	0.00019
		0.1%	R1	II	0.175	0.00213
			R2	II	0.322	0.00392
			R3	II	0.186	0.00227
	TCLP	1%	R1	II	0.053	0.00067
			R2	II	0.039	0.00049
			R3	II	0.041	0.00052

TABLE G-3. STUDY B TCLP AND EP EXTRACT ANALYSIS FOR THE  
PCE WASTE METAL CONTAMINANTS

Metal Contaminant	Test	Organic Level	Replicate Number	Extraction Fluid*/ Acid Added (ml)	Extract Concentration (mg/l)	Normalized Extracted Concentration (mg/l)
Antimony	EP	0.1%	R1	10	0.027	0.00035
			R2	10	0.028	0.00036
			R3	10	0.027	0.00035
		1.0%	R1	25	0.02	0.00028
			R2	25	0.027	0.00038
			R3	25	0.022	0.00031
	TCLP	0.1%	R1	I	0.034	0.00044
			R2	I	0.038	0.00049
			R3	I	0.038	0.00049
		1.0%	R1	I	0.039	0.00055
			R2	I	0.038	0.00053
			R3	I	0.036	0.00051
Arsenic	EP	0.1%	R1	10	<0.005	0.00006
			R2	10	0.005	0.00006
			R3	10	0.007	0.00009
		1.0%	R1	25	<0.005	0.00007
			R2	25	<0.005	0.00007
			R3	25	<0.005	0.00007
	TCLP	0.1%	R1	I	0.006	0.00008
			R2	I	0.006	0.00008
			R3	I	0.007	0.0001
		1.0%	R1	I	0.007	0.0001
			R2	I	0.006	0.00008
			R3	I		
Copper	EP	0.1%	R1	10	9.3	0.12
			R2	10	9.84	0.126
			R3	10	13.1	0.168
		1.0%	R1	25	10.7	0.150
			R2	25	11.	0.15
			R3	25	10.8	0.152
	TCLP	0.1%	R1	I	12.9	0.166
			R2	I	13.2	0.169
			R3	I	13.1	0.168

(Continued)

\* I = TCLP extraction fluid 1.

(Sheet 1 of 3)

TABLE G-3. (Continued)

Metal Contaminant	Test	Organic Level	Replicate Number	Extraction Fluid/Acid Added (ml)	Extract Concentration (mg/l)	Normalized Extracted Concentration (mg/l)
Copper (Cont.)	TCLP	1.0%	R1	I	16.5	0.232
			R2	I	16.4	0.230
			R3	I	16.1	0.226
Lead	EP	0.1%	R1	10	0.026	0.00033
			R2	10	0.03	0.0004
			R3	10	0.051	0.00065
		1.0%	R1	25	0.037	0.00052
			R2	25	0.027	0.00038
			R3	25	0.021	0.00029
		0.1%	R1	I	0.063	0.00081
			R2	I	0.064	0.00082
			R3	I	0.067	0.00086
		1.0%	R1	I	0.065	0.00091
			R2	I	0.085	0.0012
			R3	I	0.072	0.0010
	TCLP	0.1%	R1	I	0.063	0.00081
			R2	I	0.064	0.00082
			R3	I	0.067	0.00086
		1.0%	R1	I	0.065	0.00091
			R2	I	0.085	0.0012
			R3	I	0.072	0.0010
		0.1%	R1	I	0.063	0.00081
			R2	I	0.064	0.00082
			R3	I	0.067	0.00086
Silver	EP	0.1%	R1	10	<0.001	0.00001
			R2	10	<0.001	0.00001
			R3	10	0.003	0.00004
		1.0%	R1	25	0.004	0.00006
			R2	25	0.004	0.00006
			R3	25	0.003	0.00004
		0.1%	R1	I	0.009	0.0001
			R2	I	<0.001	0.00001
			R3	I	<0.001	0.00001
		1.0%	R1	I	<0.001	0.00001
			R2	I	<0.001	0.00001
			R3	I	<0.001	0.00001
	TCLP	0.1%	R1	I	0.009	0.0001
			R2	I	<0.001	0.00001
			R3	I	<0.001	0.00001
		1.0%	R1	I	<0.001	0.00001
			R2	I	<0.001	0.00001
			R3	I	<0.001	0.00001
		0.1%	R1	I	0.009	0.0001
			R2	I	<0.001	0.00001
			R3	I	<0.001	0.00001
Zinc	EP	0.1%	R1	10	23.4	0.300
			R2	10	28.1	0.361
			R3	10	36.3	0.466
		1.0%	R1	25	16.4	0.230
			R2	25	17.0	0.24
			R3	25	16.8	0.236
		0.1%	R1	I	31.6	0.406
			R2	I	32.5	0.417
			R3	I	32.5	0.417
	TCLP	0.1%	R1	I	31.6	0.406
			R2	I	32.5	0.417
			R3	I	32.5	0.417

(Continued)

(Sheet 2 of 3)

TABLE G-3. (Concluded)

Metal Contaminant	Test	Organic Level	Replicate Number	Extraction Fluid/Acid Added (ml)	Extract Concentration (mg/l)	Normalized Extracted Concentration (mg/l)
Zinc (Cont.)	TCLP	1.0%	R1	I	33.1	0.465
			R2	I	32.3	0.454
			R3	I	33.4	0.469
Barium	EP	0.1%	R1	10	0.315	0.00404
			R2	10	0.388	0.00498
			R3	10	0.442	0.00567
		1.0%	R1	25	0.321	0.00451
			R2	25	0.362	0.00508
			R3	25	0.321	0.00451
		0.1%	R1	I	0.428	0.00549
			R2	I	0.433	0.00556
			R3	I	0.517	0.00664
	TCLP	1.0%	R1	I	0.578	0.00812
			R2	I	0.564	0.00792
			R3	I	0.541	0.00760

## APPENDIX H

### GRAPHICAL REPRESENTATION OF THE RESULTS OF TCLP AND EP EXTRACTIONS FOR STUDY B METALS

Figures H-1 through H-7 are graphical representations of the TCLP and EP extractions for each metal contaminant of Study B. In these figures the normalized EP concentrations are plotted versus the normalized TCLP extract concentrations. A line with a slope of 1.0 is plotted on each graph. Points which lie on this line indicate that the extract concentrations for the EP and TCLP are equal. Points above this line indicate that the TCLP produced extracts with higher concentrations of the contaminant, and points below this line indicate that the EP resulted in extracts containing higher concentrations of the contaminants.

Figure H-5 illustrates that the lead contaminant was more aggressively extracted by the TCLP for each extraction that was performed. Figure H-6 illustrates that the WES-1.0%-mercury data plot on the y-axis. These mercury data points deviate from the majority of the average population and are suspect. Figures H-2, H-4, and H-7, which present the cadmium, chromium, and nickel data for the WES sludge, illustrate that analyses of the extracts from the WES sludge produced data with more scatter than was observed in the extracts of the other sludges.

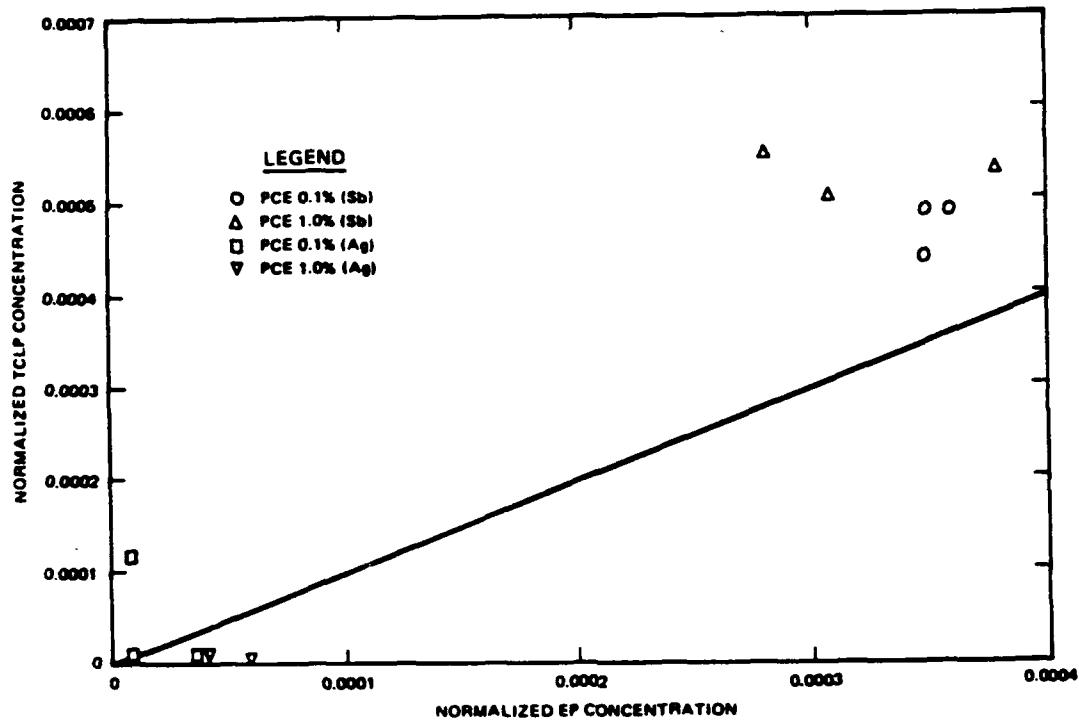


Figure H-1. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B antimony and silver contaminants.

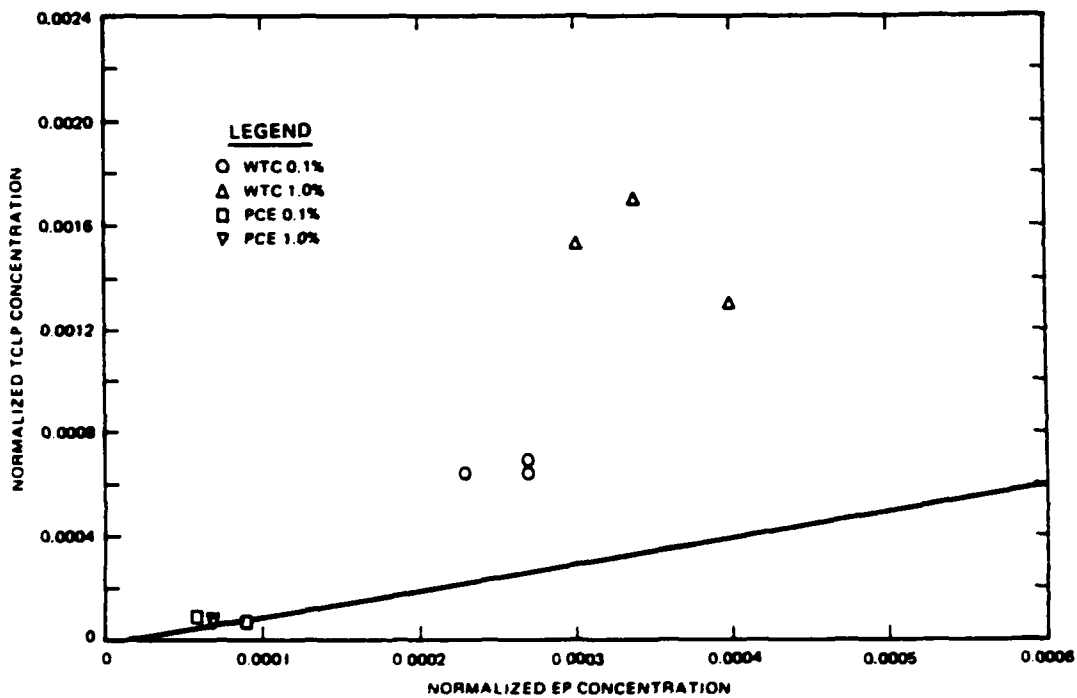


Figure H-2. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B arsenic contaminant.

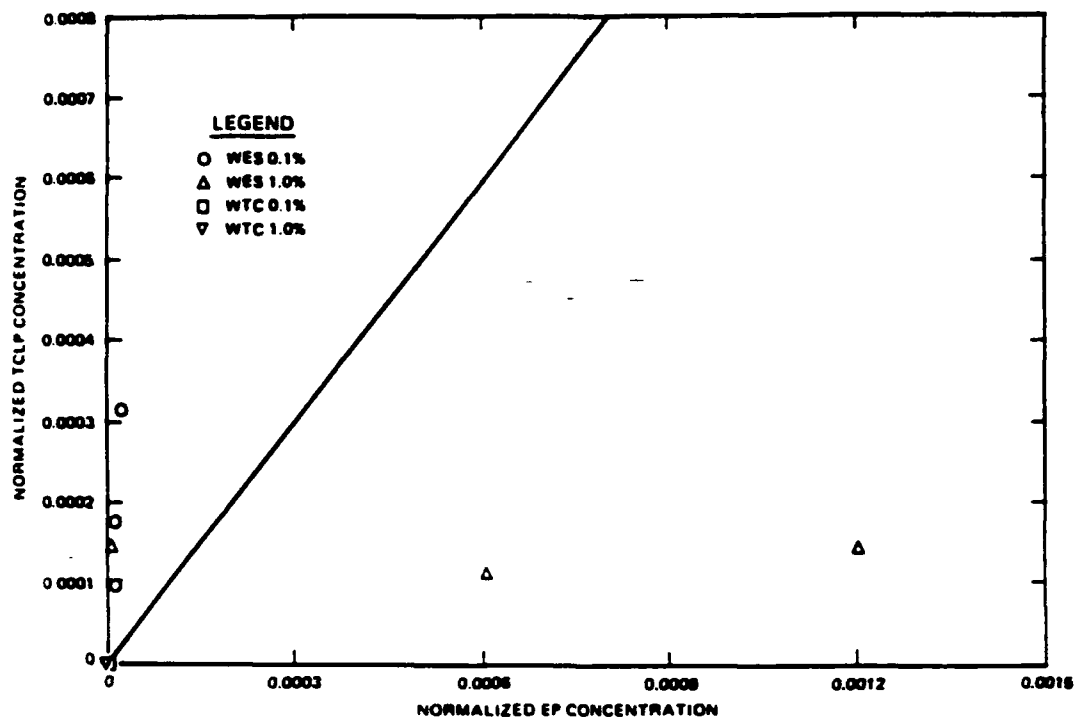


Figure H-3. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B cadmium contaminant.

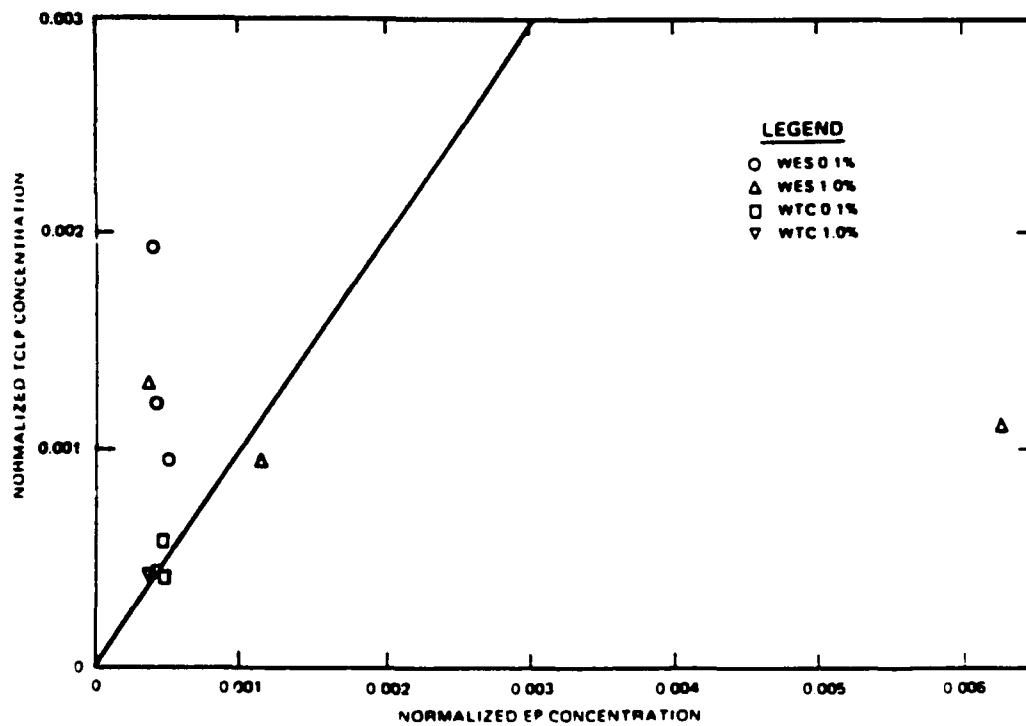


Figure H-4. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B chromium contaminant.

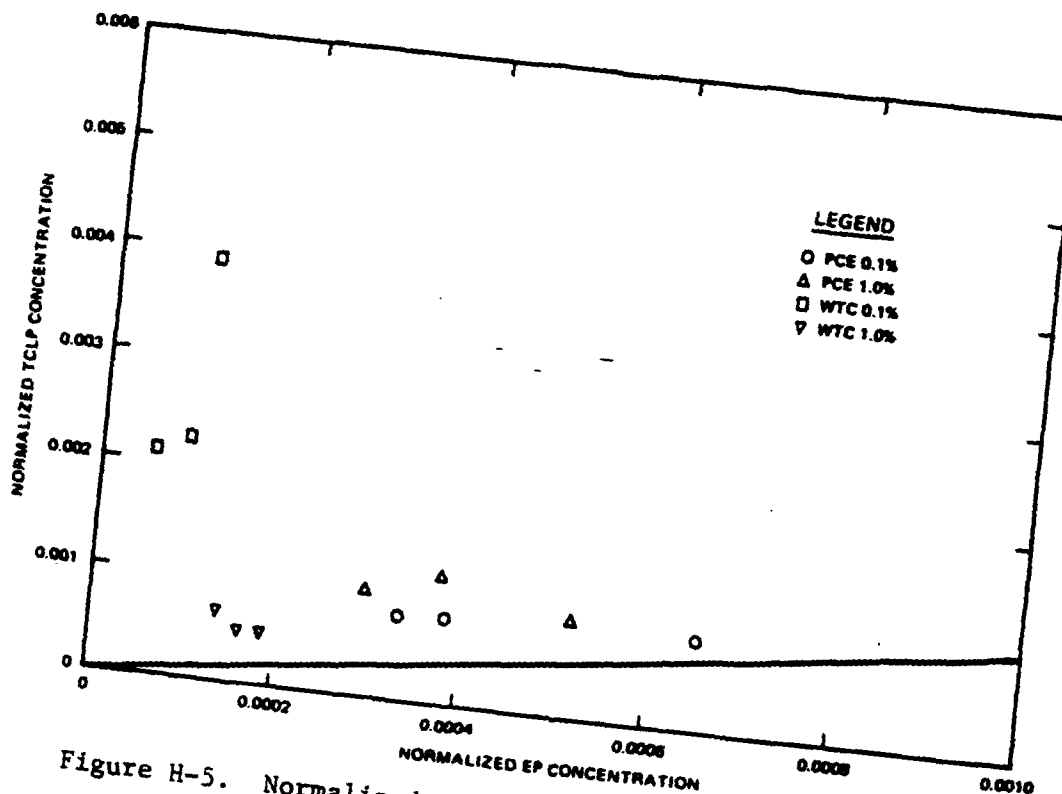


Figure H-5. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B lead contaminant.

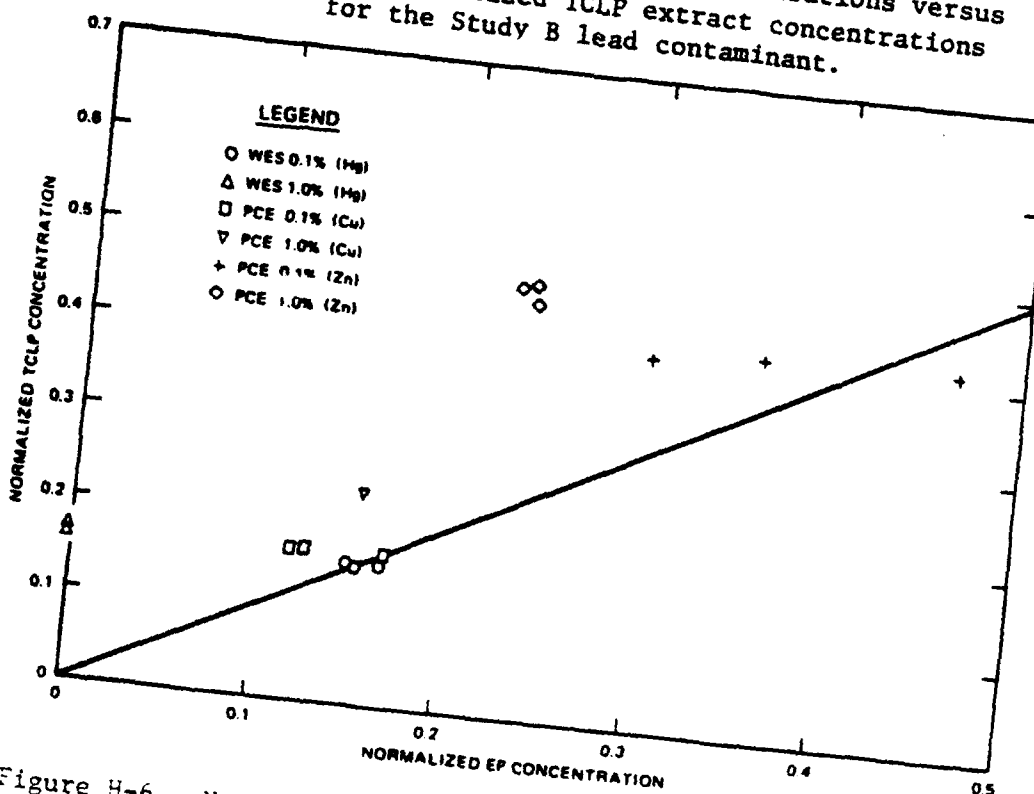


Figure H-6. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B mercury, zinc and copper contaminants.

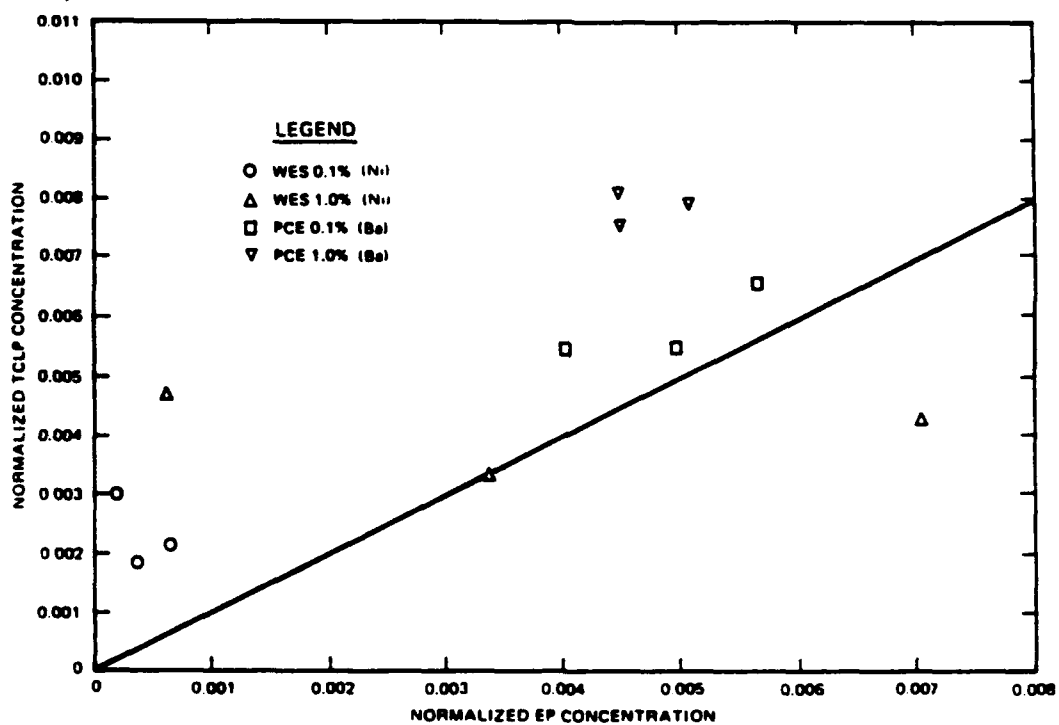


Figure H-7. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B nickel and barium contaminants.

## APPENDIX I

## STUDY B ORGANICS RAW DATA

TABLE I-1. STUDY B TCLP AND EP EXTRACT ANALYSES FOR CARBON TETRACHLORIDE

Sludge	Extraction Test	Organic Level	Replicate Number -	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	0.51	0.04
			R2	0.5	0.04
			R3	<0.25	0.02
		1%	R1	2.4	0.19
			R2	5.	0.4
			R3	4.4	0.35
WES	TCLP	0.1%	R1	1.4	0.11
			R2	0.66	0.052
			R3	0.61	0.048
		1%	R1	<5.	0.4
			R2	11.	0.88
			R3	6.8	0.55
PCE	EP	0.1%	R1	<0.5	0.03
			R2	0.11	0.0056
			R3	0.087	0.0045
		1%	R1	10.	0.6
			R2	<10.	0.6
			R3	<10.	0.6
PCE	TCLP	0.1%	R1	<0.5	0.03
			R2	<0.5	0.03
			R3	<0.5	0.03
		1%	R1	<10.	0.6
			R2	<10.	0.6
			R3	<10.	0.6
WTC	EP	0.1%	R1	<0.1	0.005
			R2	<0.1	0.005
			R3	<0.1	0.005
		1%	R1	<5.	0.3
			R2	5.	0.3
			R3	<5.	0.3
WTC	TCLP	0.1%	R1	<0.2	0.01
			R2	<0.2	0.01
			R3	<0.2	0.01
		1%	R1	<5.	0.3
			R2	<5.	0.3
			R3	<5.	0.3

TABLE I-2. STUDY B TCLP AND EP EXTRACT ANALYSES FOR CHLOROFORM

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	0.67	0.053
			R2	1.	0.08
			R3	0.96	0.075
		1%	R1	13.5	1.08
			R2	13.3	1.07
			R3	15.1	1.21
	TCLP	0.1%	R1	1.8	0.14
			R2	0.9	0.07
			R3	1.5	0.12
		1%	R1	38.8	3.11
			R2	18.	1.4
			R3	25.	2.0
PCE	EP	0.1%	R1	0.97	0.05
			R2	1.06	0.0544
			R3	1.01	0.0519
		1%	R1	20.5	1.15
			R2	23.6	1.33
			R3	27.2	1.53
PCE	TCLP	0.1%	R1	1.5	0.077
			R2	1.55	0.0796
			R3	1.62	0.0832
		1%	R1	32.5	1.83
			R2	35.4	1.99
			R3	30.2	1.70
WTC	EP	0.1%	R1	0.237	0.0116
			R2	0.22	0.011
			R3	0.221	0.0108
		1%	R1	8.16	0.410
			R2	9.48	0.477
			R3	9.29	0.467
WTC	TCLP	0.1%	R1	<0.2	0.01
			R2	<0.2	0.01
			R3	<0.2	0.01
		1%	R1	10.	0.5
			R2	8.32	0.419
			R3	9.08	0.457

TABLE I-3. STUDY B TCLP AND EP EXTRACT ANALYSES FOR 1,2-DICHLOROETHANE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	1.5	0.12
			R2	1.7	0.13
			R3	1.5	0.12
		1%	R1	36.8	2.95
			R2	35.7	2.86
			R3	43.6	3.50
WES	TCLP	0.1%	R1	1.7	0.13
			R2	1.	0.08
			R3	1.1	0.086
		1%	R1	89.1	7.15
			R2	50.	4.
			R3	45.	3.6
PCE	EP	0.1%	R1	3.57	0.183
			R2	3.66	0.188
			R3	3.59	0.184
		1%	R1	53.4	3.00
			R2	57.6	3.24
			R3	60.9	3.42
PCE	TCLP	0.1%	R1	4.	0.2
			R2	4.26	0.219
			R3	4.43	0.227
		1%	R1	70.4	3.95
			R2	73.8	4.15
			R3	70.	4.
WTC	EP	0.1%	R1	0.81	0.039
			R2	0.735	0.0358
			R3	0.735	0.0358
		1%	R1	45.5	2.29
			R2	43.6	2.19
			R3	46.	2.3
WTC	TCLP	0.1%	R1	0.633	0.0308
			R2	0.442	0.0215
			R3	0.392	0.0191
		1%	R1	47.2	2.37
			R2	43.2	2.17
			R3	42.3	2.13

TABLE I-4. STUDY B TCLP AND EP EXTRACT ANALYSES FOR  
1,1,1-TRICHLOROETHANE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	0.92	0.072
			R2	1.1	0.086
			R3	0.87	0.068
		1%	R1	16.9	1.36
			R2	18.4	1.48
			R3	19.7	1.58
WES	TCLP	0.1%	R1	2.7	0.21
			R2	1.4	0.11
			R3	1.7	0.13
		1%	R1	58.4	4.68
			R2	39.	3.1
			R3	43.	3.4
PCE	EP	0.1%	R1	0.45	0.023
			R2	0.62	0.032
			R3	0.59	0.030
		1%	R1	12.	0.67
			R2	14.2	0.798
			R3	19.	1.1
PCE	TCLP	0.1%	R1	1.2	0.062
			R2	1.22	0.0626
			R3	1.18	0.0606
		1%	R1	24.6	1.38
			R2	25.8	1.45
			R3	24.8	1.39
WTC	EP	0.1%	R1	0.306	0.0149
			R2	0.287	0.0140
			R3	0.286	0.0139
		1%	R1	13.4	0.674
			R2	16.5	0.830
			R3	15.3	0.770
WTC	TCLP	0.1%	R1	0.563	0.0274
			R2	0.457	0.0223
			R3	0.34	0.017
		1%	R1	29.5	1.484
			R2	22.9	1.15
			R3	22.1	1.11

TABLE I-5. STUDY B TCLP AND EP EXTRACT ANALYSES FOR TRICHLOROETHENE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	3.4	0.27
			R2	3.8	0.30
			R3	3.2	0.25
		1%	R1	56.9	4.56
			R2	67.3	5.40
			R3	69.7	5.59
WES	TCLP	0.1%	R1	8.6	0.67
			R2	5.2	0.41
			R3	6.9	0.54
		1%	R1	153.	12.3
			R2	120.	9.6
			R3	130.	10.
PCE	EP	0.1%	R1	1.41	0.0724
			R2	1.75	0.0898
			R3	1.27	0.0652
		1%	R1	28.7	1.61
			R2	34.1	1.92
			R3	38.4	2.16
PCE	TCLP	0.1%	R1	4.8	0.25
			R2	2.88	0.148
			R3	2.94	0.151
		1%	R1	37.4	2.10
			R2	39.2	2.20
			R3	43.3	2.43
WTC	EP	0.1%	R1	2.46	0.120
			R2	2.17	0.106
			R3	2.33	0.114
		1%	R1	94.2	4.74
			R2	98.	4.9
			R3	102.	5.13
WTC	TCLP	0.1%	R1	2.73	0.133
			R2	2.58	0.126
			R3	2.33	0.114
		1%	R1	147.	7.39
			R2	130.	6.5
			R3	130.	6.5

TABLE I-6. STUDY B TCLP AND EP EXTRACT ANALYSES FOR BENZENE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	1.5	0.12
			R2	1.7	0.13
			R3	1.6	0.13
		1%	R1	37.	3.0
			R2	44.	3.5
			R3	47.9	3.84
WES	TCLP	0.1%	R1	2.9	0.23
			R2	1.7	0.13
			R3	2.3	0.18
		1%	R1	98.	7.9
			R2	81.	6.5
			R3	77.	6.2
PCE	EP	0.1%	R1	2.63	0.135
			R2	2.92	0.150
			R3	2.3	0.12
		1%	R1	43.2	2.43
			R2	56.1	3.15
			R3	63.2	3.55
PCE	TCLP	0.1%	R1	5.6	0.29
			R2	5.07	0.260
			R3	5.21	0.267
		1%	R1	72.8	4.09
			R2	77.5	4.35
			R3	79.4	4.46
WTC	EP	0.1%	R1	0.946	0.0461
			R2	0.874	0.0426
			R3	0.902	0.0440
		1%	R1	53.5	2.69
			R2	55.6	2.80
			R3	56.6	2.85
WTC	TCLP	0.1%	R1	0.88	0.043
			R2	0.812	0.0396
			R3	0.686	0.0334
		1%	R1	71.8	3.61
			R2	58.7	2.95
			R3	56.7	2.85

TABLE I-7. STUDY B TCLP AND EP EXTRACT ANALYSES FOR 1,1,2,2-TETRACHLOROETHANE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	<0.25	0.020
			R2	<0.25	0.020
			R3	<0.25	0.020
		1%	R1	<1.	0.08
			R2	<1.	0.08
			R3	<1.	0.08
WES	TCLP	0.1%	R1	<0.25	0.020
			R2	<0.2	0.02
			R3	<0.2	0.02
		1%	R1	<5.	0.4
			R2	<5.	0.4
			R3	<5.	0.4
PCE	EP	0.1%	R1	7.74	0.397
			R2	7.31	0.375
			R3	6.87	0.353
		1%	R1	95.7	5.38
			R2	90.6	5.09
			R3	91.8	5.16
PCE	TCLP	0.1%	R1	8.8	0.45
			R2	9.06	0.465
			R3	9.26	0.475
		1%	R1	65.6	3.68
			R2	78.9	4.43
			R3	94.4	5.30
WTC	EP	0.1%	R1	<0.1	0.005
			R2	<0.1	0.005
			R3	<0.1	0.005
		1%	R1	<5.	0.3
			R2	<5.	0.3
			R3	<5.	0.3
WT	TCLP	0.1%	R1	<0.2	0.01
			R2	<0.2	0.01
			R3	<0.2	0.01
		1%	R1	<5.	0.3
			R2	<5.	0.3
			R3	<5.	0.3

TABLE I-8. STUDY B TCLP AND EP EXTRACT ANALYSES FOR TETRACHLOROETHENE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	2.7	0.21
			R2	3.6	0.28
			R3	3.	0.2
		1%	R1	27.9	2.24
			R2	27.8	2.23
			R3	22.2	1.78
	TCLP	0.1%	R1	8.5	0.67
			R2	4.6	0.36
			R3	7.9	0.62
		1%	R1	37.	3.0
			R2	40.	3.
			R3	39.	3.1
PCE	EP	0.1%	R1	3.25	0.167
			R2	3.1	0.16
			R3	2.74	0.141
		1%	R1	28.5	1.60
			R2	26.7	1.50
			R3	29.7	1.67
PCE	TCLP	0.1%	R1	3.3	0.17
			R2	3.09	0.159
			R3	3.17	0.163
		1%	R1	12.2	0.685
			R2	13.6	0.764
			R3	14.3	0.803
WTC	EP	0.1%	R1	1.08	0.0526
			R2	0.922	0.0449
			R3	1.01	0.0492
		1%	R1	17.	0.86
			R2	20.	1.
			R3	19.6	0.9860
WTC	TCLP	0.1%	R1	1.66	0.0809
			R2	1.6	0.078
			R3	1.55	0.0755
		1%	R1	39.9	2.01
			R2	39.7	2.00
			R3	40.	2.

TABLE I-9. STUDY B TCLP AND EP EXTRACT ANALYSES FOR TOLUENE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	2.8	0.22
			R2	3.3	0.26
			R3	3.	0.2
		1%	R1	55.3	4.44
			R2	58.2	4.67
			R3	52.8	4.28
WES	TCLP	0.1%	R1	5.4	0.42
			R2	2.9	0.23
			R3	5.	0.4
		1%	R1	100.	8.
			R2	92.	7.4
			R3	89.	7.1
PCE	EP	0.1%	R1	1.78	0.0914
			R2	1.28	0.0657
			R3	1.06	0.0544
		1%	R1	35.	1.97
			R2	35.9	2.02
			R3	39.1	2.20
PCE	TCLP	0.1%	R1	2.5	0.13
			R2	2.47	0.127
			R3	2.52	0.129
		1%	R1	31.3	1.76
			R2	36.5	2.05
			R3	39.5	2.22
WTC	EP	0.1%	R1	1.36	0.0663
			R2	1.12	0.0546
			R3	1.23	0.0599
		1%	R1	62.8	3.16
			R2	68.3	3.44
			R3	65.9	3.31
WTC	TCLP	0.1%	R1	1.5	0.073
			R2	1.34	0.0653
			R3	1.34	0.0653
		1%	R1	94.2	4.74
			R2	91.2	4.59
			R3	83.3	4.19

TABLE I-10. STUDY B TCLP AND EP EXTRACT ANALYSES FOR ETHYLBENZENE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	4.6	0.36
			R2	5.5	0.43
			R3	5.7	0.45
		1%	R1	35.	2.8
			R2	33.6	2.70
			R3	32.9	2.64
WES	TCLP	0.1%	R1	11.	0.86
			R2	16.	1.3
			R3	25.	2.0
		1%	R1	46.	3.7
			R2	52.	4.2
			R3	44.	3.5
PCE	EP	0.1%	R1	2.09	0.107
			R2	2.24	0.115
			R3	1.77	0.0909
		1%	R1	35.4	1.99
			R2	33.5	1.88
			R3	34.7	1.95
PCE	TCLP	0.1%	R1	2.3	0.12
			R2	2.37	0.122
			R3	2.32	0.119
		1%	R1	20.9	1.17
			R2	20.6	1.16
			R3	21.3	1.20
WTC	EP	0.1%	R1	3.17	0.154
			R2	2.7	0.13
			R3	2.92	0.142
		1%	R1	35.	1.8
			R2	36.1	1.82
			R3	37.2	1.87
WTC	TCLP	0.1%	R1	3.74	0.182
			R2	3.85	0.188
			R3	4.24	0.207
		1%	R1	80.7	4.06
			R2	127.	6.39
			R3	79.1	3.98

TABLE I-11. STUDY B TCLP AND EP EXTRACT ANALYSES FOR 2-BUTANONE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	47.6	3.73
			R2	42.8	3.36
			R3	17.	1.3
		1%	R1	171.	13.7
			R2	181.	14.5
			R3	212.	17.0
WES	TCLP	0.1%	R1	23.	1.8
			R2	14.	1.1
			R3	14.	1.1
		1%	R1	280.	22.
			R2	300.	24.
			R3	190.	15.
PCE	EP	0.1%	R1	5.46	0.280
			R2	5.33	0.274
			R3	4.77	0.245
		1%	R1	132.	7.41
			R2	137.	7.70
			R3	131.	7.36
PCE	TCLP	0.1%	R1	4.7	0.24
			R2	5.18	0.266
			R3	6.28	0.322
		1%	R1	132.	7.41
			R2	135.	7.58
			R3	136.	7.64
WTC	EP	0.1%	R1	9.65	0.470
			R2	10.9	0.531
			R3	8.21	0.400
		1%	R1	156.	7.85
			R2	180.	9.05
			R3	153.	7.70
WTC	TCLP	0.1%	R1	5.98	0.291
			R2	6.37	0.310
			R3	6.52	0.318
		1%	R1	167.	8.40
			R2	166.	8.35
			R3	164.	8.25

TABLE I-12. STUDY B TCLP AND EP EXTRACT ANALYSES FOR  
4-METHYL-2-PENTANONE

Sludge	Extraction Test	Organic Level	Replicate Number	Extract Concentration (mg/l)	Normalized Extraction Concentration (mg/kg)
WES	EP	0.1%	R1	60.	5.
			R2	50.	4.
			R3	14.	1.1
		1%	R1	175.	14.0
			R2	193.	15.5
			R3	210.	17.
WES	TCLP	0.1%	R1	17.	1.3
			R2	11.	0.86
			R3	12.	0.94
		1%	R1	350.	28.
			R2	290.	23.
			R3	300.	24.
PCE	EP	0.1%	R1	12.5	0.642
			R2	12.4	0.637
			R3	10.	0.51
		1%	R1	236.	13.3
			R2	227.	12.8
			R3	236.	13.3
PCE	TCLP	0.1%	R1	11.	0.56
			R2	10.3	0.529
			R3	10.6	0.544
		1%	R1	220.	12.
			R2	263.	14.8
			R3	258.	14.5
WTC	EP	0.1%	R1	7.66	0.373
			R2	8.3	0.40
			R3	7.06	0.344
		1%	R1	315.	15.8
			R2	278.	14.0
			R3	301.	15.1
WTC	TCLP	0.1%	R1	4.64	0.226
			R2	5.17	0.252
			R3	4.84	0.236
		1%	R1	297.	14.9
			R2	333.	16.8
			R3	288.	14.5

## APPENDIX J

### GRAPHICAL REPRESENTATION OF THE RESULTS OF TCLP AND EP EXTRACTIONS FOR STUDY B ORGANICS

Figures J-1 through J-12 are graphical representations of the TCLP and EP extractions for each organic contaminant of Study B. In these figures the normalized EP extract concentrations are plotted versus the normalized TCLP extract concentrations. A line with a slope of 1.0 is plotted on each graph. Points which lie on this line indicate that the extract concentrations for the EP and TCLP are equal. Points above this line indicate that the TCLP produced extracts with higher concentrations of the contaminant, and points below this line indicate that the EP resulted in extracts containing higher concentrations of the contaminants.

To compare Figures J-1 through J-12, the difference in scales must be considered. To maintain reasonable resolution, the scales were not equivalent for any of the organic contaminants evaluated.

Inspection of Figures J-1 through J-12 illustrates that for most contaminants with organic concentration level of 0.1%, the EP and TCLP data were grouped closely around the line near the axis. This indicates that the TCLP and the EP produce extracts of almost equal concentrations. All the contaminants with organic concentration levels of 1.0%, except 1,1,2,2,-tetrachlorethane (Figure J-6) and tetrachloroethene (Figure J-7), were more aggressively extracted by the TCLP than the EP.

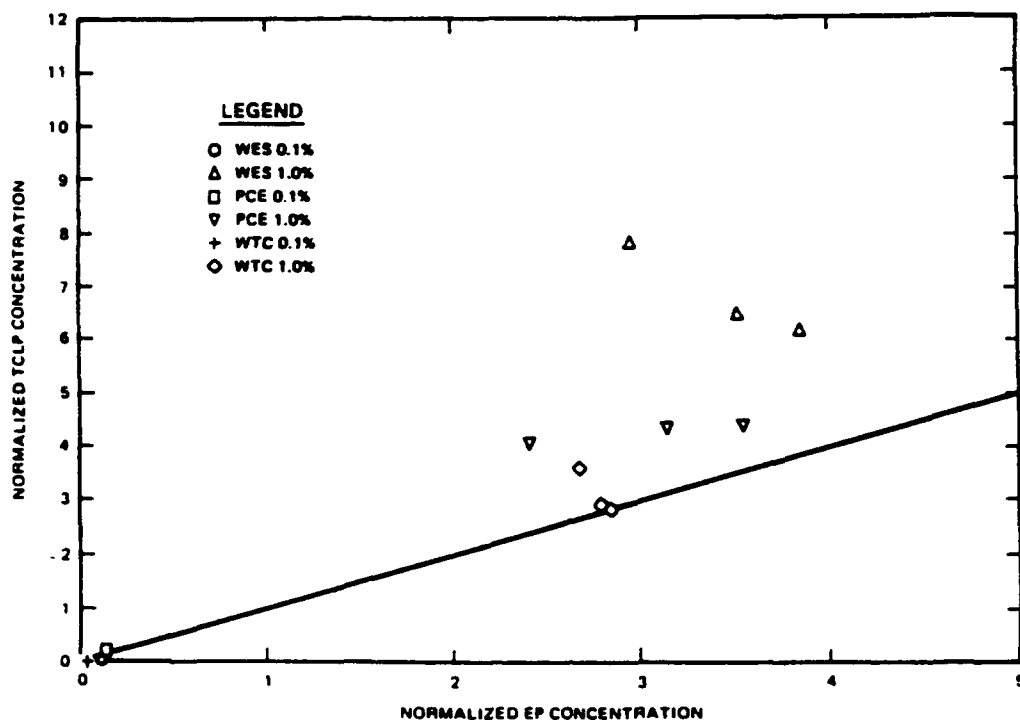


Figure J-1. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B benzene contaminant.

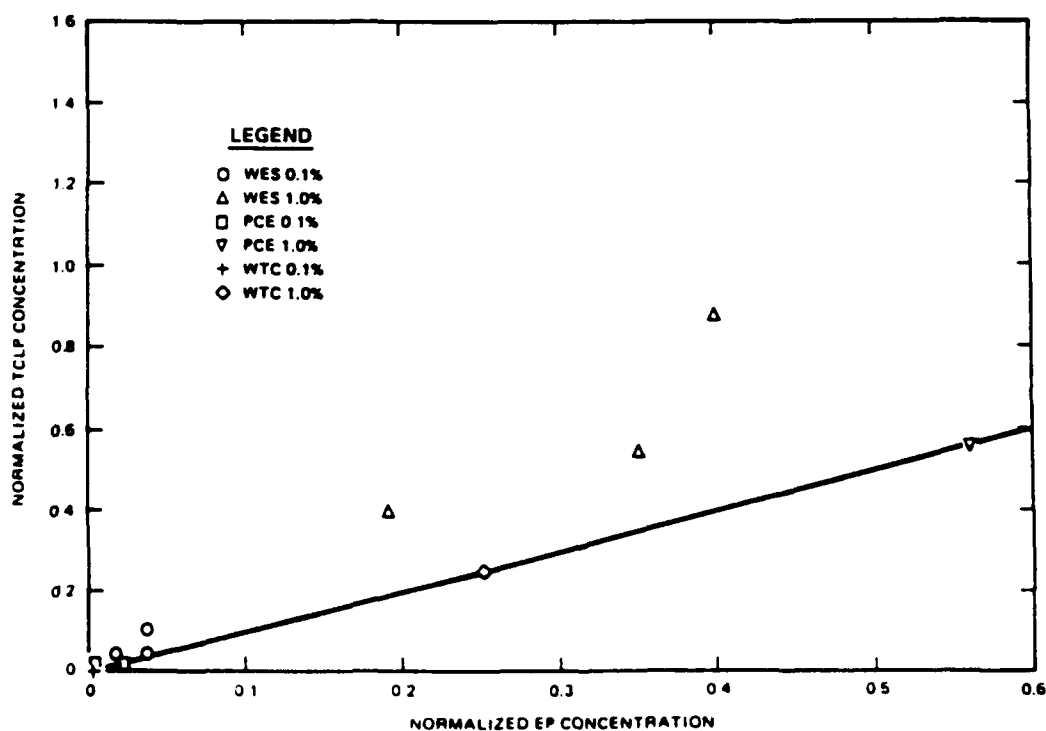


Figure J-2. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B carbon tetrachloride contaminant.

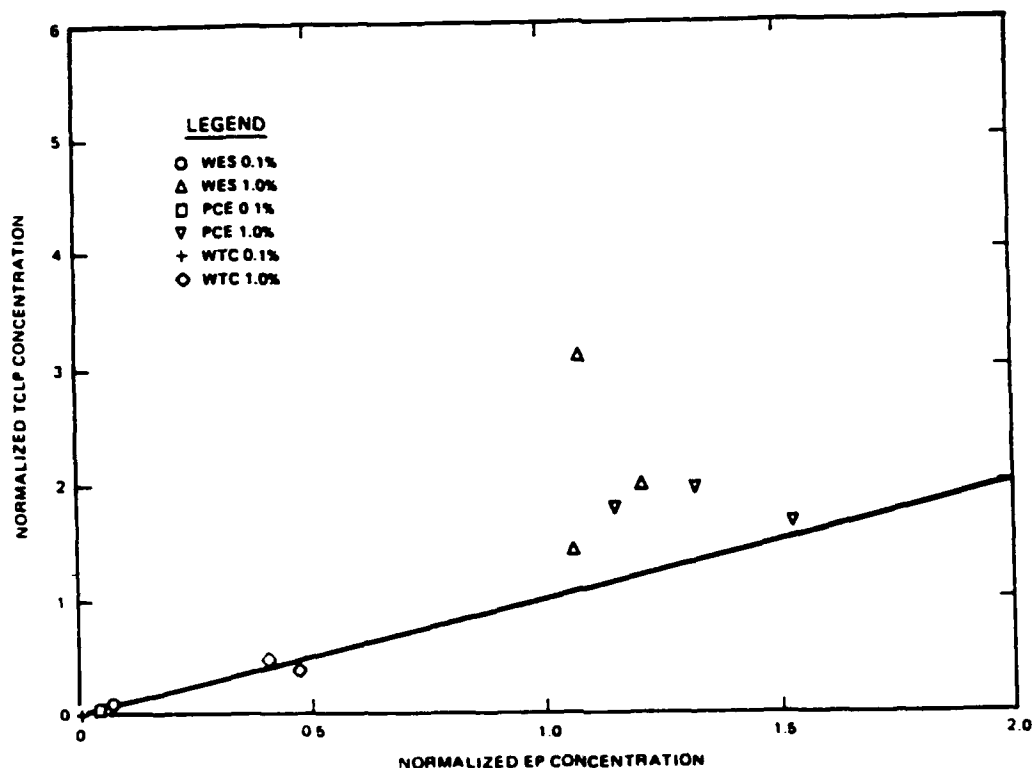


Figure J-3. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B chloroform contaminant.

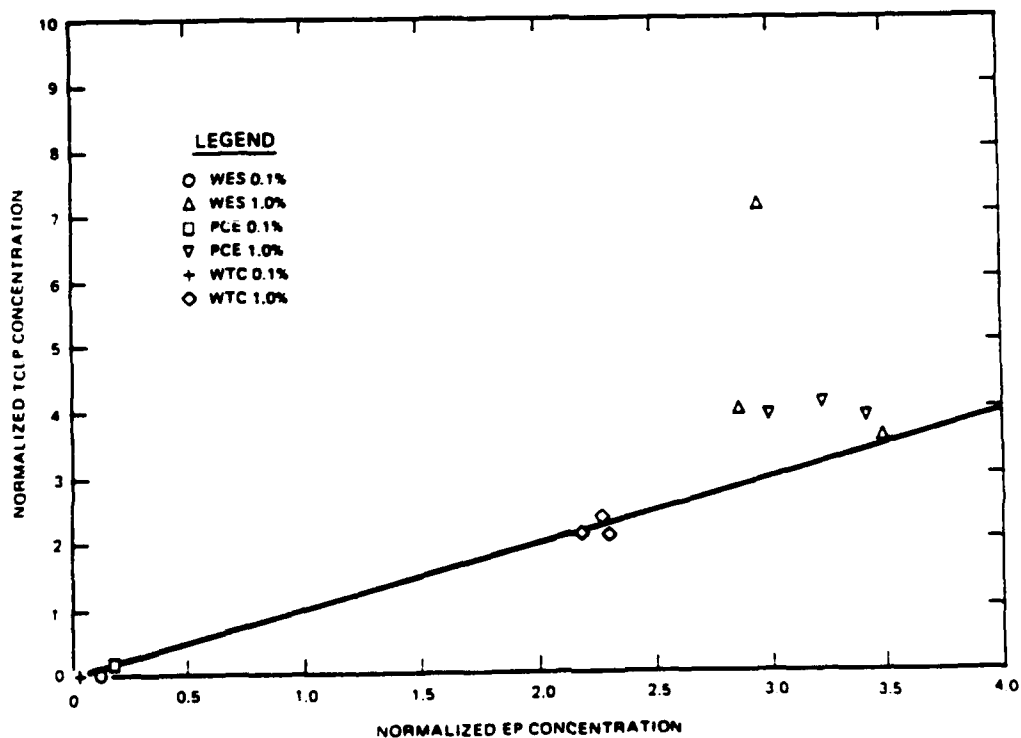


Figure J-4. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B 1,2-dichloroethane contaminant.

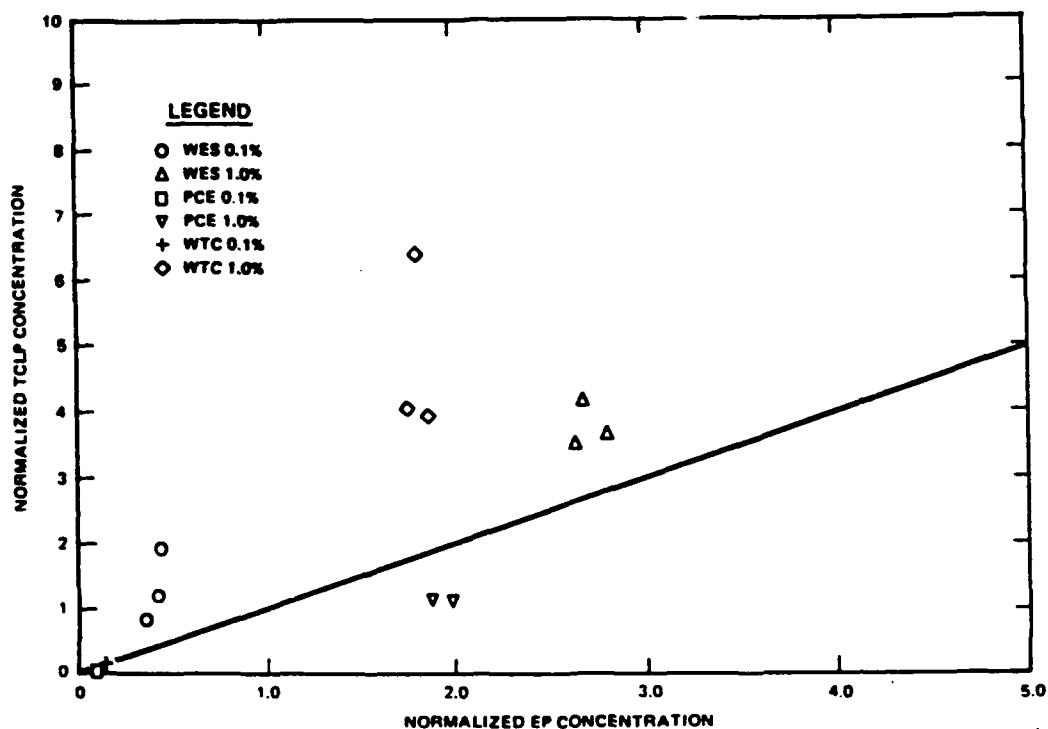


Figure J-5. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B ethylbenzene contaminant.

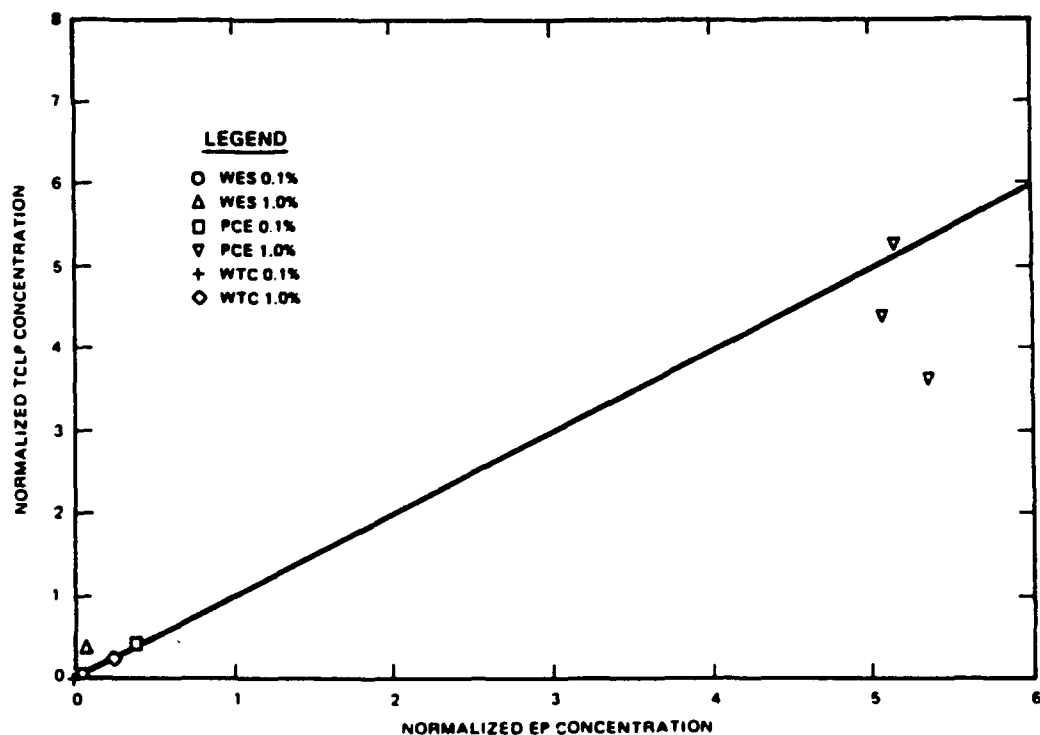


Figure J-6. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B 1,1,2,2-tetrachloroethane contaminant.

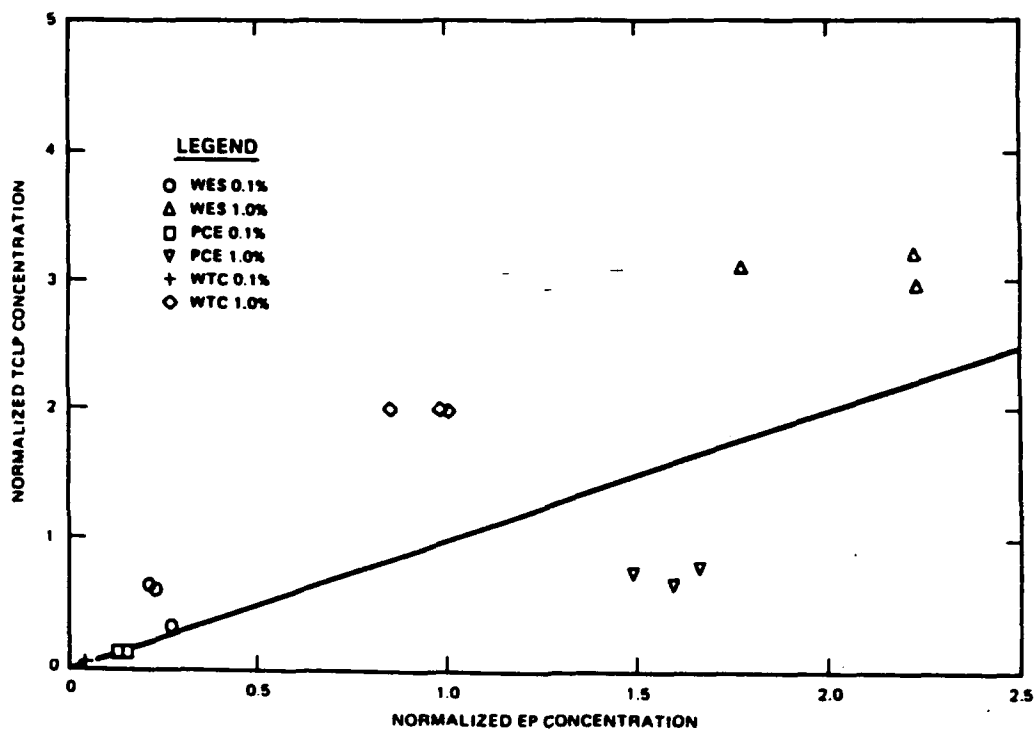


Figure J7. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B tetrachloroethene contaminant.

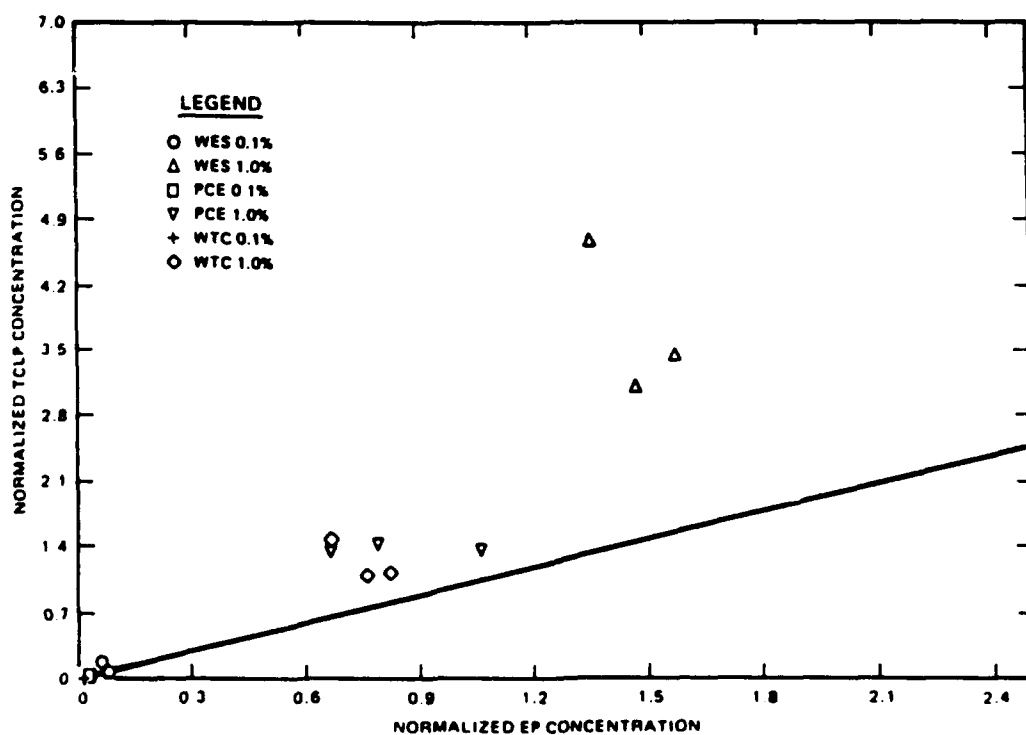


Figure J8. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B 1,1,1-trichloroethane contaminant.

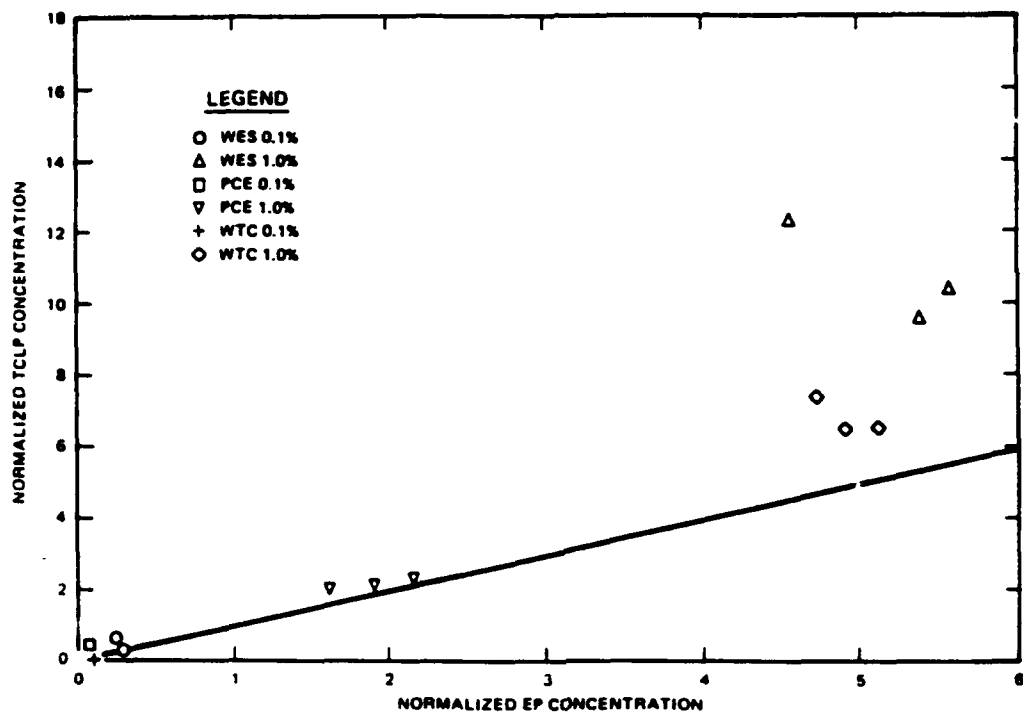


Figure J-9. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B trichloroethene contaminant.

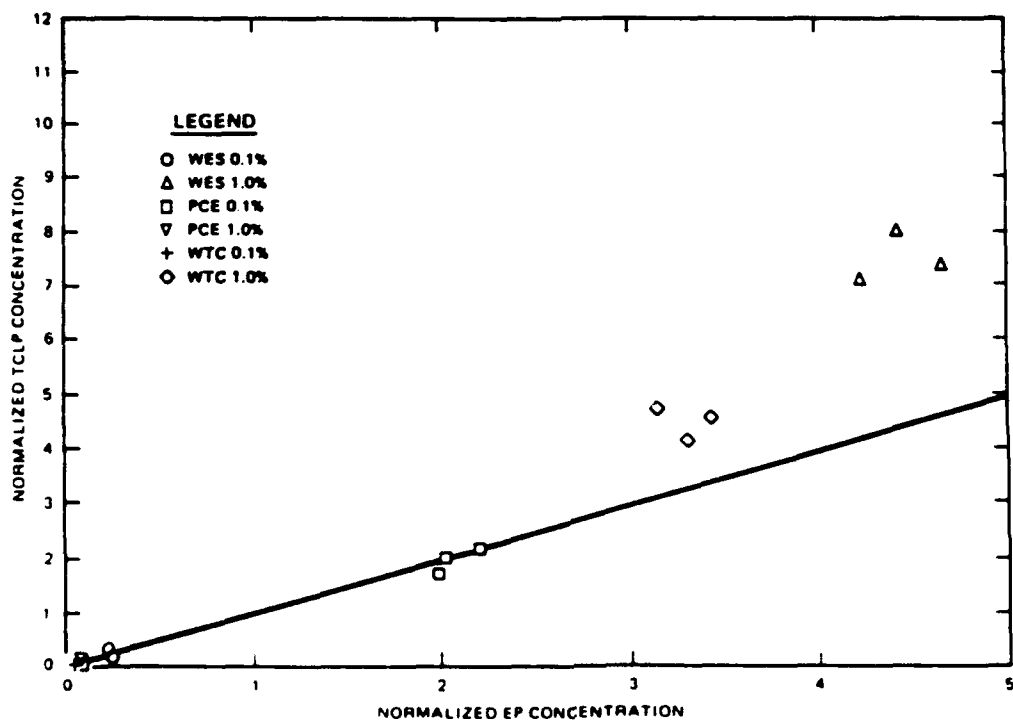


Figure J-10. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B toluene contaminant.

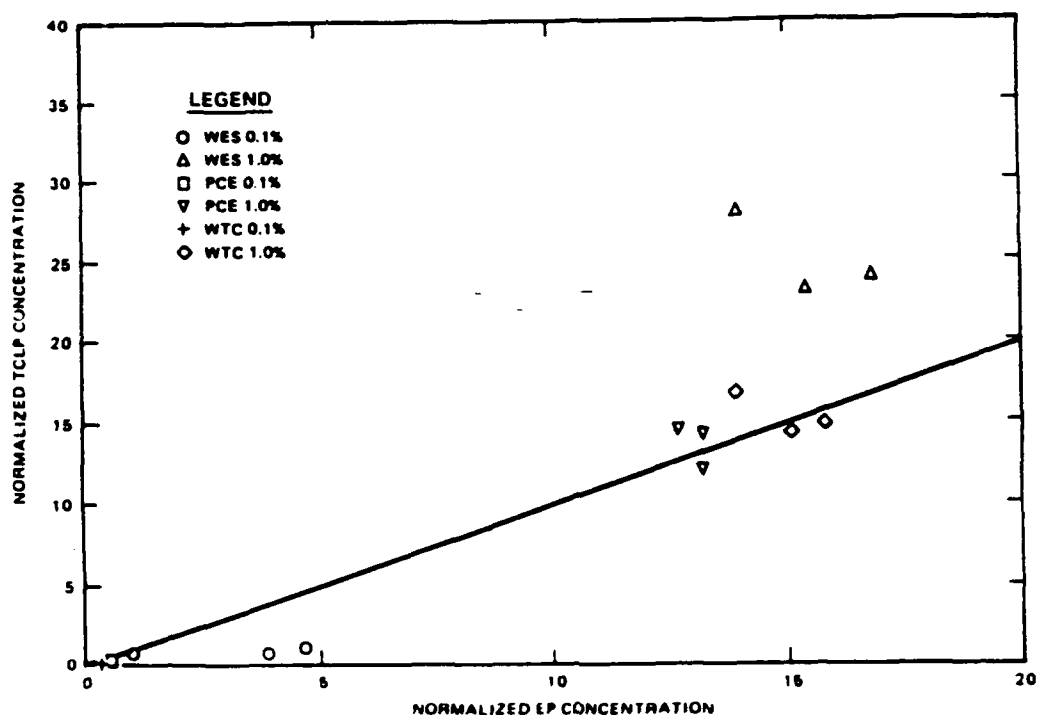


Figure J-11. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B 4-methyl-2-pentanone contaminant.

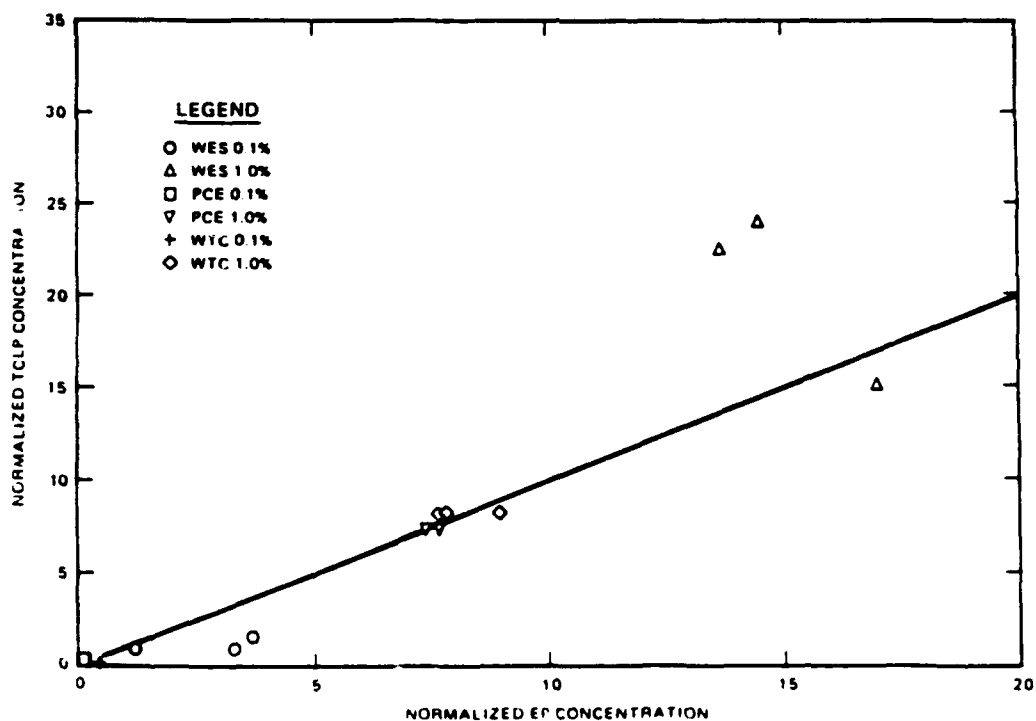


Figure J-12. Normalized EP extract concentrations versus the normalized TCLP extract concentrations for the Study B 2-butanone contaminant.

### **Waterways Experiment Station Cataloging-In-Publication Data**

Bricka, R. Mark.

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TA7 W34 no.EL-92-33